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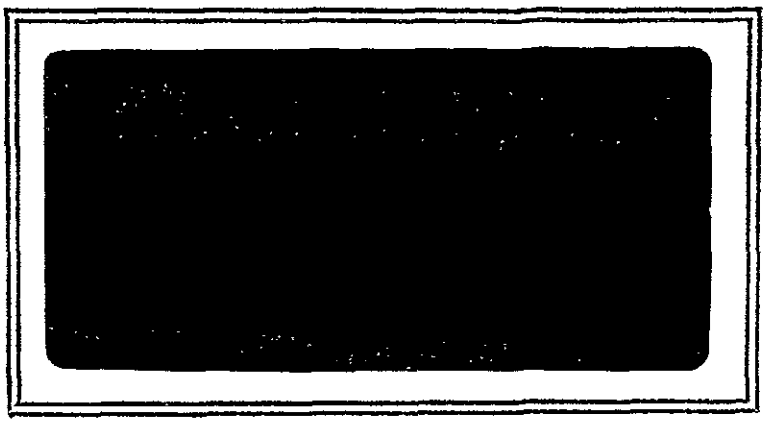

THE ELECTRIC STORAGE BATTERY CO.

THE CARL F. NORBERG RESEARCH CENTER

YARDLEY, PENNA.

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HEAT STERILIZABLE IMPACT RESISTANT CELL DEVELOPMENT

JET PROPULSION LABORATORY
CONTRACT NO. 951296

REPORT FOR FIRST QUARTER, 1969
JANUARY 1 TO MARCH 31, 1969

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Prepared by: G. F. NORDBLOM, Section Leader
A. M. CHREITZBERG, Assistant Director of Engrg.

Approved by: G. W. BODAMER, Program Director
G. S. HARTMAN, Director of Engineering

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ABSTRACT

Further tests conducted during the first quarter of 1969 have shown that volatile components in the epoxy system may be responsible for low capacity as well as cell leakage through crazing during sterilization. A vacuum treatment to remove MEK from the epoxy system is proposed to alleviate crazing and a heating cycle prior to the final seal is beneficial for good capacity.

The embrittlement of negative silver grids by mercury is a problem area for high impact cells. While this study is just beginning, observations indicate that damage which occurs during the first few months may be self healing on standing about a year. A barrier layer of lead plated on the grid did not solve the problem.

Work has progressed on cycling prior to heat sterilization, and a method of discharge has been proposed to prepare the cell for this purpose.

Since the cells in this program should function after about nine months of interplanetary travel, there was a question as to whether they should be on stand or float during this interval. Fortunately, there were available sealed cells which had been on stand or float for about two years. These were discharged and cycled to determine their condition. The data show that cells on float-charge maintained their capacity whereas those on stand lost their capacity and that it could not be regained even after a subsequent recharge.

Development of wet heat sterilizable 5 and 25 AH cells capable of 4,000 "g" impact is complete through design review and construction of four 5 AH cells for shock tests at JPL. Charge efficiency of zirconium reinforced positives is 17-21% lower than expected; however, no gassing is observed during wet heat sterilization and strength of composite electrode appears to be adequate at 4,000 "g" in all axis but terminals forward. Chemically etched massive pure Ag grids will support negatives safely when supported in slots molded into the cell jar walls. This support reduces apparent negative plate area by 31% and voltage by 7% (1.48 volts to 1.38 volts per cell) at the C/2 rate of discharge.

ABSTRACT (continued)

Medium cycle life 25 AH cells have been designed and manufactured in five designs to evaluate the effects of two negative active material densities, two separator systems, and two wraps. On the formation cycle after 72 hours heat sterilization at 135°C output energy densities ranged from 44 to 50 WH/lb of cell with discharge plateau voltages of 1.49 - 1.51 volts per cell. Cycling tests on 10 hour charge (2 hour discharge at 50% depth are in progress. Forty cells are in production for delivery to JPL during April and May 1969.

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ELECTROCHEMISTRY

I. INTRODUCTION

The problem of sealed cells was studied quite intensively during the period January to March 1969. The two main areas studied were the effects of organic constituents on cell capacity and the problem related to cell leakage. Cell leakage is associated with the crazing of the epoxy sealant during sterilization. The data suggest that the ratio of resin to catalyst is not critical provided most of the MEK solvent is removed. Furthermore, experiments with Novolak DEN438/EK85 alone or with DMP30 alone, or with Catalyst 11 alone (each material in the annular space between Teflon insert and nickel bomb) showed either some total capacity loss or poorer capacity at the high rate of discharge. Such observations indicate that the exposure of glue lines to water vapor or KOH solution should be minimized as far as possible.

The examination of cells which had completed four cycles and had stood for about three months revealed that the silver grids had been made friable by Hg. Although grids which have stood for longer periods do not show this weakened condition, some studies have been carried out to determine the nature and the mechanism of the grid damage. As a possible means of surmounting the problem, several negative grids were lead plated before the negative mix was added to them. The data for cells made with these lead plated grids when combined with other data on lead plated silver wires indicate that lead plating of the grids will not solve the problem.

One of the problems common to battery design is the requirement for all cells to be very similar in charge-discharge characteristics. If the charge-discharge characteristics are significantly different, overcharging of some cells may occur. If the battery is sealed, large pressures will be developed in the system leading to battery case rupture. In an attempt to ensure the selection of cells with similar performance, cells subjected to one or more charge-discharge cycles before sterilization were examined. The results appear to depend on (1) bake out procedure prior to the formation cycle, (2) the state of charge of the cell, and (3) the method of ensuring a completely discharged cell.

Finally, the work on long cycle life has been started. Data from float and stand cells were collected and gave information on separator and on methods of maintaining a cell. The data show that cells on float-charge maintained their capacity while those on stand lost their capacity which could not be regained even after subsequent recharge.

II. EFFECTS OF ORGANIC COMPONENTS ON CELLS

Ever since it was observed that the capacity of a sterilized Ag-ZnO cell was affected by the presence of organic constituents especially the epoxy

used to seal the cell, a number of experiments have been conducted to find an adequate sealant. Among the parameters suspected as contributing to the epoxy effect, was the ratio of resin to catalyst. In addition to having no detrimental effect on cell capacity, a sealant must provide an adequate seal even when the cell is subjected to high temperature (about 135°C). The cells constructed with the various epoxy sealants, namely DEN438-DMP30, DEN438-Catalyst 11, and Allaco's All Bond have consistently leaked especially at the terminals. In the case of the DEN EK85/DMP30 sealant, it seemed reasonable to assume that for thick layers of sealant the total amount of MEK trapped in the sealant should be larger. As the amount of MEK in the sealant increased the greater should be the tendency toward crazing, unless the MEK was somehow removed. Thus, the epoxy was subjected to a vacuum to remove most of the volatile MEK. Cells constructed using decreased MEK concentrations should not craze and hence should maintain a good seal. Unfortunately, unless cells were overpotted, leakage occurred.

The epoxy sealant may affect the cell capacity in two ways. One way which is direct is the effect of the epoxy on the cell performance due to a chemical interaction with the electrode system. This effect which has been mentioned in several earlier reports leads to a loss of capacity and will be discussed later. The other way by which epoxy affects cell capacity is to its inability to produce a good case-to-cover seal.

A higher or lower capacity than expected may be obtained when a cell leaks. This may be explained as follows: In the case where cell capacity should be decreased by a material in the vapor phase within the cell, it is obvious that if the cell leaks, the material could escape and hence lead to higher capacity than expected. On the other hand, loss of electrolyte due to a poor seal can be a cause for low cell capacity. Examination of the data in Table I reveals some of the above conclusions. For example, the better average performance of cells 43-1, 43-2, and 46-2 compared to that of cells 51-1, 51-2, and 51-3 can be accounted for by poor sealing of the 40 series cells. The 51 series cells were overpotted and hence should not leak; whereas, the 40 series were not overpotted and leaks occurred at the terminal wires. The 53 series cells were constructed in a manner similar to the 51 series cells but the material was held under a vacuum to remove excess MEK. Thus, it seems as though excess MEK unless removed would reduce cell capacity through chemical interaction with the electrode system. The resin to catalyst ratio does not appear to be critical.

The effect of epoxy as well as other organics on cell capacity has been studied quite extensively both in regular cell cases and in specially designed Teflon inserts. These studies and in particular those studies conducted in nickel bombs with a specially designed Teflon insert indicate that it is possible to cycle cells in the presence of epoxy and other organics without reducing cell capacity. The results may be best explained on the basis of

TABLE I

Performance of Ag-Zn Cells Cycled Before Sterilization as Related to Epoxy Sealant

Cycle	Cell Capacity (amp-hrs/g Ag)								
	40-1	43-1	43-2	46-2	51-1	51-2	51-3	53-1	53-2
Resin/Catalyst Ratio *	100/5	100/3	100/5	100/6	100/3	100/5	100/7	100/2	100/2
Formation Charge	0.33	0.35	0.33	0.36	0.40	0.41	0.40	0.38	0.39
Formation Discharge	0.32	0.33	0.31	0.35	0.36	0.39	0.35	0.37	0.37
Sterilization - 72 hrs at 135°C									
First Discharge	0.23	0.27	0.27	0.29	0.13	0.32	0.21	0.36	0.34
Second Discharge	0.24	0.25	0.28	0.28	0.17	0.33	0.23	0.36	0.36
Third Discharge	0.25	0.27	0.29	0.32	0.15	0.33	0.22	0.36	0.36
Fourth Discharge	0.25	0.31	0.31	-	-	-	-	-	--

* DEN438/DMP30

the size of the head space of the cell. In the regular PPO cases where the effects of the organic constituents on cell capacity have occurred the head space is much smaller than in the bombs with Teflon inserts where the cell capacity is not affected. The entire work with the Teflon inserts is summarized in Table II. Because of special construction, the experiments in the Teflon inserts could not be conducted at a head space that was as limited as that obtainable in regular PPO case. Thus, limited head space in Table II refers to a condition whereby the free space was reduced as much as possible but not to the same extent as in PPO cases.

Except for DMP30 the data for various components tested in Teflon cases indicate that the components do not reduce the cell capacity as evidenced by values of about 0.35 amp-hr/g Ag. However, the voltage at which the capacity is obtained is affected. The ratio of first to second stage discharge capacity is about 5.00/1.5 for normal cells. As shown in Table III, the ratios for Catalyst 11 are 2.8/3.9 and 3.3/3.3, although the total capacity was 0.39 AH/g Ag. As might be expected, there seems to be a correlation between the cell impedance and the first to second stage discharge ratio. The initial value of 0.09 ohms compared with the fourth cycle value of 0.03 ohms for the impedance of the cells containing Catalyst 11 is in the direction of the improved first to second stage discharge ratio. The decrease in resistance might be caused by the breaking down of surface films and the observation merits further study.

Also shown in Table III are the output/input ratio, midvolts, and pressure changes. For the first cycle, the output to input ratios for DMP30 and DEN 438/EK85 were normal whereas the ratio for Catalyst 11 was somewhat lower than normal. Very little pressure change occurred during the various cycles and hence any problems due to high pressure may be ignored for DMP 30 and DEN438/EK85. However, the pressure change for cells containing Catalyst 11 was unusually high for a cell with so much head space and suggests that a pressure problem with Catalyst 11 might be expected.

III. EFFECT OF MERCURY ON THE STRENGTH OF NEGATIVE GRIDS

For some time now, it has been questioned whether or not the silver grid in cells with mercury in the zinc electrode was weakened by amalgamation. Recent findings indicate that the grids taken from cells which had been cycled and stood for about 3 months were friable whereas grids which stood around for a year or more were not friable. This result is not surprising inasmuch as it is well known that initially mercury enters the grain boundaries but eventually diffuses into the grain. A number of studies are now underway in an effort to further understand the nature and mechanism of the grid damage. One possible way of surmounting the grid damage might be to coat the silver grid of the negative electrode with lead. Four cells were constructed in nickel bombs and sterilized at 135°C for 72 hours Two of the cells had zinc electrodes which had 0.0011 in. of lead plate on the silver grids, and the other two had standard zinc electrodes and functioned

TABLE II

Summary of the Sterilization Effects of Organic Compounds on Cell Capacity

(Experiments in Teflon Inserts in Nickel Bombs)

Excess Head Space	Capacity (AH/g Ag) 1st Dischg.		2nd Dischg.		3rd Dischg.		4th Dischg.	
	A	B	A	B	A	B	A	B
(1) GX, 476 Unster.	.34	.32	.33	.30	.33	.31	.34	.30
(2) GX, 476 Ster. *	.33	.33	.33	.32	.35	.35	.36	.36
Limited Head Space								
(3) GX alone. Unster.	.39	.40	.35	.39	.37	.37	.37	.37
(4) GX alone Ster.	.39	.24	.39	.26	.39	.30	-	.36
(4a) " " "	.35	.36	.32	.37	.35	.35	-	-
(4b) " " "	.38		-		-		-	
(5) GX, 531-801 ster.	.34	.33	.36	.35	.37	.35	-	-
(6) GX, cured DEN.../ DMP30 Ster.	.35	.40	.35	.35	.35	.37	-	-
(7) GX alone; cell heated at 160°F for 16 hrs. in N_2 before activa. and ster.	.36	-	.35	-	-	-	-	-
(8) GX, 534-801 ster.	.35	.34	.35	.34	.35	.35	.35	.33
(8a) " " "	.39	.40	-	-	-	-	-	-
(9) GX, DEN etc. cured in activated and sealed bomb before ster.	.35	.34	.35	.34	.35	.33	.34	.32
(10) RAI-110 alone ster.	.37	.39	.37	-	.37	-	.38	-
(11) RAI-116 alone ster.	.36	.40	.35	-	.37	-	.36	-
(12) GX, PPO, and uncured epoxy ster.	.40	.39	-	-	-	-	-	-
(13) GX, cell heated at 160°C for 16 hrs. in air before activa. and ster.	.40	.40	-	-	-	-	-	-
(14) GX, with no allowance for expansion, ster.	.40	.40	-	-	-	-	-	-

TABLE II (continued)
Summary of the Sterilization Effects of Organic Compounds on Cell Capacity
(Experiments in Teflon Inserts in Nickel Bombs)

Limited Head Space	Capacity (AH/g Ag) 1st Dischg.		2nd Dischg.		3rd Dischg.		4th Dischg.	
(15) GX, DMP30 alone ster.	.35	.31	.33	.31	.34	.27	.36	.27
(16) GX, DEN438 EK85 alone ster.	.37	.38	.35	.38	.35	.34	.35	.31
(17) GX, Catalyst 11 alone ster.	.37	.37	.39	.39	.40	.40	.40	.40
(18) GX, EM476 alone ster.	.38	.38	.37	.38	.37	.37	.37	.36

TABLE III

Effects of Cell Components on Capacity, Voltage and Pressure of Cells Sealed Then Sterilized

	DMP-30		DEN438/EK85		Catalyst 11	
	1	2	1	2	1	2
Cycle #1						
Discharge capacity						
1st stage (AH)	3.90	4.84	5.76	5.40	2.80	3.32
" " 2nd "	1.60	1.44	1.01	1.25	3.92	3.33
AH/gram Ag	0.31	0.35	0.38	0.37	0.39	0.39
Midvolt	1.42	1.40	1.39	1.40	1.37	1.37
Pressure change (psi)	+1.5	0	-1.4	0	+6.1	-
Output/Input (%)	81	85	82	84	72	76
AC Impedance (ohm)	0.03	0.03	0.03	0.03	0.087	0.089
Cycle #2						
Discharge capacity						
1st stage	4.08	5.34	5.90	5.70	4.78	5.40
" " 2nd "	0.28	0.64	0.93	0.88	2.24	1.68
AH/gram Ag	0.31	0.33	0.38	0.37	0.39	0.39
Cycle #3						
Discharge Capacity						
1st stage	4.32	5.04	5.50	5.42	5.04	5.08
" " 2nd "	0.36	1.07	0.60	0.83	2.13	2.07
AH/gram Ag	0.27	0.34	0.34	0.35	0.40	0.40
Cycle #4						
Discharge Capacity						
1st stage	4.10	6.02	5.26	5.50	6.40	6.42
" " 2nd "	0.62	0.49	0.28	0.38	0.75	0.88
AH/gram Ag	0.27	0.36	0.31	0.33	0.40	0.40
Midvolt	1.45	1.43	1.43	1.42	1.40	1.40
Pressure change	+1.0	-0.5	+1.0	0	0	-
Output/Input	100	83	94	105	104	97
AC Impedance	-	-	-	-	0.031	0.030

as controls. Each zinc electrode mix contained 4% mercury. Because this work was just started, the data will be reported in the next quarter. However, the results from cells made with lead plated grids when combined with other data on lead plated silver wires indicate that lead plating of the grids will not solve the problem.

IV. STERILIZATION OF CYCLED CELLS

The ability to construct a battery from sealed cells requires that the cells must have almost identical charge-discharge characteristics. If the cells vary significantly in their charge acceptance or discharge capacity, the battery will become unbalanced leading to evolution of gas and high cell pressure. Increased cell pressure puts an extra strain on the case-to-cover seal and could lead to cell failure. One way of matching cells is to actually cycle the cells and make the selection before constructing the battery. However, because of the requirement that the final battery must be sterilized, it became necessary to examine the feasibility of sterilizing cycled cells. The results appear to depend on (1) bake out procedure prior to formation cycle, (2) the state of charge of the cell, and (3) the method of ensuring a completely discharged cell.

A. Resterilization of Sealed Cells

To see what was involved in sterilizing sealed cells after one or more qualification cycles and specifically whether the metallic mercury present in the negatives would limit the capacity of the silver electrodes (mercury effect), ten good cells which had completed at least four cycles were selected for resterilization. These cells had been used to determine the effect on capacity of organic components used in the construction of sealed cells. Since all materials except epoxy was without effect, these cells were well suited to the resterilization experiment.

To prepare the cells for resterilization they were discharged to zero volts at five amperes and shorted. They were then resterilized at 135°C for 68 hours. The original component under test and other pertinent cycle data are shown in Table IV. The results show that the capacity of half of the cells was adversely affected whereas that of the other half was improved by resterilization. It would seem that the mercury effect is not involved directly or there would be no capacity improvement for any of the cells. Obviously, there is a need for a thorough study of preparing cycled cells for sterilization.

B. The Effects of Sterilization on Sealed Cells

In working with cells which were sterilized in the unformed condition it was observed that the steps involved in processing prior to sterilization affected cell behavior in later life. It had been determined that a heat cycle after activation but before sealing was beneficial, and the assumption

TABLE IV

Effects of the Second Sterilization of Cells on Capacity, Voltage, and Pressure of Cells Which Had Been Sealed and Sterilized Once Before

	PPO 534	PPO 534	RAI 110	RAI 116	RAI 110	RAI 116	PPO + Epoxy	GX alone	EM 476	EM 476
Net Charge Cap. (1)	6.18	6.12	6.86	6.56	7.60	7.42	6.82	9.13	6.73	6.63
" " " (2)	5.48	5.09	6.77	6.84	6.72	7.52	7.77	7.37	6.93	6.69
Midvoltage (1)	1.48	1.45	1.42	1.40	1.40	1.42	1.45	1.45	1.43	1.43
" (2)	1.46	1.46	1.40	1.42	1.39	1.41	1.45	1.44	1.45	1.45
Total Dischg. Cap. (1)	6.16	5.89	6.70	6.45	7.20	6.62	5.89	6.85	6.60	6.44
" " " (2)	5.24	4.39	6.20	6.39	6.22	6.87	6.84	7.13	6.81	6.52
Output/Input (1)	100%	95%	98%	98%	95%	89%	86%	75%	98%	97%
" " (2)	96%	86%	92%	94%	93%	92%	88%	97%	99%	98%
AH/gram Ag Dischg. (1)	.35	.33	.38	.38	.40	.37	.32	.37	.37	.36
" " " " (2)	.30	.25	.35	.36	.35	.38	.38	.39	.38	.37
Days of stand between cycles	104	104	104	104	71	71	71	71	4	4
Pressure Change due to 2nd ster.	none	none	-0.4psi	-0.4psi	-0.4psi	*	-1.0psi	none	none	-0.2psi

(1) Before Second Sterilization
(2) After Second Sterilization

* Not available

was made that it eliminated from the cell harmful volatile ingredients whose source was the epoxy sealant. In the experiments to follow changes in the heat cycle were investigated. In cells of the type prepared for Tables V and VI it is beneficial to have a heat cycle immediately after preformation (reduction of mercury) while the cell is open whether or not a prior heat cycle had been done. As seen from Tables V and VI the highest capacity is obtained when the "B" stage temperature of the epoxy is limited to 45°C and the cure temperature is 100°C. The cure heat cycle should be after preformation for maximum capacity.

The conditions of preparation for the cells in Tables V and VI and the results are as follows: For all cells 8 layers of separator were used. Electrolyte concentration was 42% potassium hydroxide by weight before dissolving 100 gm/l of zinc oxide, after which its specific gravity was 1.49. Separator thickness allowance per layer of separator was 0.003 in. Silver electrodes for the cells of Table V were of the low density variety, while those of Table VI were of the higher density. No significance is attached to this difference, since in prior cells containing low density silver, good performance was obtained. All cells contained three silver electrodes and four of zinc oxide (sintered Teflon). The outside negative electrodes contained 4 gms of mix in each and the inside ones either 6 or 7 depending on the silver electrodes used.

The following observations of performance before sterilization follow from Table V: (1) higher load voltages result from cells having been exposed to 45°C compared to 65°C before activation; (2) better capacities resulted when the heat cycle temperature was 100°C rather than 80°C or no heat cycle as for cell 20-1; (3) variation in load voltages related to heat cycle temperature was not consistent; (4) performances of all cells of Table V were poorer then expected for unsterilized cells, and were poorer than for similar cells, similarly sealed, and sterilized before formation.

Performances of all seven cells after sterilization were poorer than before. Since cells 26-1 and 27-1 lost considerable electrolyte as a result of the leaks mentioned above, deterioration in their performance was anticipated. Even so, cell 27-1 was best of the seven.

The next group of cells, shown in Table VI, were processed in the same manner as was cell 27-1 except that the heat cycle followed preformation instead of preceding it. This change apparently caused the difference between good and bad performance both prior to and following sterilization. First cycle capacity values after sterilization were approximately 5 to 7% below comparable values obtained before sterilization. During the second cycle their values increased so that they were 0 to 2% below their pre-sterilization values. Voltages are considered satisfactory for this separator system.

TABLE V

Study of Silver-Zinc Cells Cycled Before Sterilization

Cell Number	21-1	21-2	26-1	27-1	20-1	20-2	20-3
Primary Seal	All Bond	All Bond	All Bond	All Bond	All Bond	All Bond	All Bond
Temperature for "B" Stage	65°C	65°C	45°C	45°C	65°C	65°C	65°C
Cure Temperature	80°C	100°C	80°C	100°C	Ambient	80°C	100°C
Cure (before, after) conversion of 323-43	Before	Before	Before	Before	Before	Before	Before
Hours to convert 323-43, theoretical	13.8	13.8	13.8	13.8	13.8	13.8	13.8
Hours to convert 323-43, actual	13.4	16.4	25.0	25.3	16.0	28.2	18.1
Grams of active material, silver	14.7	14.7	14.7	14.7	14.7	14.7	14.7
Grams of negative mix (91-3-6)	20	20	20	20	20.0	20.0	20.0
Before sterilization:							
Formation charge, amp-hrs	4.53	5.41	5.60	5.97	5.72	5.50	5.56
Formation discharge,							
amp-hrs, total	3.53	4.41	4.50	4.95	4.53	4.45	4.91
amp-hrs/gm Ag	0.240	0.30	0.306	0.336	0.308	0.302	0.334
90 min. voltage	1.364	1.357	1.387	1.397	1.329	1.365	1.355
After sterilization:							
Charge - amp-hrs, Total	2.89	3.62	3.61	4.33	2.59	3.93	4.64
First dischg.							
amp-hrs, total	2.38	2.85	3.10	4.08	1.94	3.34	3.48
amp-hrs/gm Ag	0.161	0.193	0.210	0.277	0.132	0.226	0.277
Discharge, amp-hrs, total	2.94	3.29	3.17	3.93	3.49	4.12	
Second dischg.							
am-hrs, total	2.13	3.07	2.87	3.95	3.41	3.84	
amp-hrs/gm Ag	0.144	0.209	0.195	0.260	0.232	0.261	
Recharge, amp-hrs, total	2.65	3.27		4.32		3.50	3.62
Third dischg,							
amp-hrs, total	2.60	3.13		4.02		3.54	3.82
amp-hrs/gm Ag	0.176	0.212		0.273		0.240	0.260

TABLE VI

Study of Silver-Zinc Cells Cycled Before Sterilization

Cell Number			29-1	35-1	35-2
Primary seal			All bond	All bond	All bond
Temperature for "B" stage			45°C	45°C	45°C
Cure temperature			100°C	100°C	100°C
Cure (before, after) conversion of 323-43			After	After	After
Hours to convert 323-43, theoretical			15.1	15.1	15.1
Hours to convert 323-43, actual			16.5	16.0	16.0
Grams of active material, silver			17	17	17
Grams of negative electrode mix (91-3-6)			22	22	22
Before Sterilization					
Formation charge - ampere hours			7.05	7.18	7.14
Formation discharge -	amp-hrs, Total		6.88	6.65	6.91
	amp-hrs/gm Ag		0.402	0.390	0.40
	90 min. voltage		1.428	1.410	1.439
After Sterilization					
Charge - Total			6.80	6.90	7.05
First discharge -	Total amp. hrs.		6.52	6.25	6.62
	amp-hrs/gm Ag		0.383	0.368	0.389
	90 min. voltage		1.413	1.398	1.421
Recharge - Total			6.56	6.47	6.63
Second discharge,	amp-hrs, Total		6.75	6.66	6.85
	amp-hrs/gm Ag		0.397	0.391	0.403
	140 min. voltage		1.418	1.389	1.408

Unresolved is the question of whether the additional heat cycle before preformation is necessary. This will be investigated in future studies. Nonetheless, it is clear that preparing cells for sterilization after cycling involves attaining the proper state of charge. There are probably many choices of load resistors possible. Generally, for the cells included here, a 100 ohm resistor was placed across the cell terminals following the normal discharge and it was allowed to remain until the voltage decayed to a value within the range of 80 to 100 mv. Following an open circuit interval, the cell voltage should be within the range of 0.75 to 0.85 volts at the start of sterilization. Since these cells have a nominal rating of 4 amp-hrs, the corresponding resistor value for any cell can be obtained from the equation $R = \frac{400}{C}$ where R is the resistance in ohms and C is the capacity in amp-hrs.

At this point, matching of cells before the final sterilization seems hopeful but has not been completely demonstrated.

V. LONG CYCLE LIFE CELLS

One of the requirements for the long cycle life cells of Task X is that they withstand 400 cycles at 50% depth of discharge after nine months of interplanetary travel. Whether cells should stand or float during this time has been a question. Fortunately, some of the first sealed and sterilized cells were put on stand or float after the fourth cycle (Report for Second Quarter 1967, JPL Contract 951296, p. 5, 14; Fourth Quarter 1967, p. 19). Recently these cells were removed from stand and float for further testing.

A. Stand-Float Performance of Some Sterilized Cells

A group of cells which had been sterilized at 135°C for 120 hours, and then cycled four times were placed either on stand or were floated at a constant potential of 1.96 volts. All but one of the cells were on the float-stand regime for about 2 years. The one exception was constructed 6 or 7 months later. The data are shown in Tables VII, VIII, and IX. The results indicate the feasibility of constructing a sealed then sterilized cell capable of delivering good capacity ($\approx$ 0.35 AH/g Ag) after 24 months of float charge. Therefore, these cells should be adequate for a mission as long as nine months.

The first seven cells were sealed in PPO cases before sterilization and examination of the case failed to reveal any high pressure within the cells. The most important observation, however, is that even after 2 years the cells on the float regime had a capacity of about 0.35 AH/g Ag. On the other hand, the cells on stand had poor capacity (< 0.2 AH/g Ag) and failed to improve when cycled further. Some attention should be given to the fact that on the discharge following the long float only 50 to 65% of the capacity was obtained on the first stage. However, on the following cycle the cells behaved normally. Perhaps a discharge then a charge cycle immediately before the batteries arrive at their destination may be beneficial.

TABLE VII
Construction of Stand and Float Cells (1)

Cell No.	* 4-116	12	11	10	9	8	7	Control #1	Rep. #3
Date of Const.	5/11/67	4/25/67	4/25/67	4/25/67	4/25/67	4/25/67	4/25/67	6/6/67	12/1/67
No. of Plates	5	5	5	5	5	5	5	5	7
Separator (5 layers)	116	110	110	110	110	110	110	110	SWRI-GX
Treatment	ster.	ster.	ster.	ster.	ster.	ster.	ster.	unster.	unster.
Weight of Ag (grams)	11.80	11.77	12.08	12.05	11.93	12.11	11.89	12.10	17.46

* Wrapped Pos., 40% KOH

(1) Ster. at 135°C for 120 hrs. in 43% KOH containing 115 gms of ZnO/liter.

TABLE VIII

Cycle Data Prior to Stand or Float

Cell #	4-116		12		11		10		9	
	C	D	C	D	C	D	C	D	C	D
Cycle #1	5.22	3.75	4.80	2.75	3.87	2.70	4.94	3.80	2.88	1.65
" #2	3.36	3.33	2.15	2.16	2.76	2.30	3.55	3.15	2.02	1.62
" #3	-	3.25	2.19	1.95	2.69	2.70	3.34	3.13	2.29	2.20
" #4	3.21	3.10	1.92	1.76	2.73	2.50	3.03	2.68	2.42	2.21

	8		7		Control #1		Rep. #3 Total		Stage 1	
	C	D	C	D	C	D	C	D	C	D
Cycle #1	3.36	3.15	5.55	4.20	4.65	3.50	6.46	4.45	6.46	4.45
" #2	2.86	2.58	3.96	3.52	3.74	3.76	4.62	4.97	4.62	4.97
" #3	2.70	2.50	-	3.46	-	4.05	7.04	5.48	6.03	5.48
" #4	2.56	2.50	3.34	3.13	3.73	3.60	6.89	5.57	5.87	5.57

Note: C = Charge (AH)

D = Discharge

TABLE IX
Capacities (AH) of Stand and Float Cells

Cell No.	F 4-116	S 12	F 11	S 10	F 9	S 8	F 7	F Control #1	F Rep. #3
Charge Acceptance Before Stand or Float	2.97	1.91	2.61	2.67	2.42	2.54	2.99	3.41	5.96
1st stage dischg at 100 mA/in^2	2.52	0.13	2.92	0.17	2.44	0.12	2.84	3.58	5.53
2nd stage dischg at 20 ma/in^2	2.38	0.20	1.62	0.50	1.58	1.17	1.17	0.99	0.26
Total Dischg. Cap.	4.90	0.33	4.54	0.67	4.02	1.29	4.01	4.57	5.79
AH/gm of Ag	0.41	0.03	0.38	0.05	0.34	0.11	0.34	0.38	0.33
1st stage charge at 7 ma/in^2	3.73	1.88	3.48	1.79	3.07	1.17	3.07	3.48	6.72
20% DOD at 100 ma/in^2	0.75	0.38	0.69	0.36	0.61	0.23	0.61	0.69	1.34
2nd stage charge at 5.6 ma/in^2	1.54	0.50	1.37	0.48	1.16	0.51	1.29	1.37	0.14
Net charge cap.	4.52	2.01	4.15	1.91	3.61	1.44	3.74	4.25	5.52
1st stage dischg. at 100 ma/in^2	3.33	1.62	3.90	1.62	3.42	0.32	3.66	3.66	3.55
2nd stage dischg. at 20 ma/in^2	1.19	0.45	0.69	0.53	0.66	1.20	0.46	0.92	0.19
Total dischg. cap.	4.52	2.07	4.59	2.15	4.08	1.52	4.12	4.58	3.74
AH/gm of Ag	0.38	0.18	0.38	0.18	0.34	0.13	0.35	0.38	0.21

It is of interest to note that even though the float cells had accepted about 2.7 AH net charge prior to float, they were able to deliver about 4.5 AH after float charge. The reason for this is not clear, but would indicate that the Ag electrode was utilized to a greater extent and this may be equivalent to the familiar two stage charge. The charge acceptance capacity of about 3.5 AH on the recycle indicates a permanent improvement of the cell.

Finally, the apparent low capacities of the cells before the long stand and float is somewhat misleading. The capacity reported before the long float and stand regime was a single step discharge. Had a second stage discharge been run the capacity might well have been equal to the capacity obtained after the long float regime.

Because the float cells have acceptable capacity they will be life cycled at 50% DOD and then dissected. The cells that were on stand along with one of the cells that were on float will be taken apart and the components analyzed.

B. Cells for Cycle Life Determination

For cycle life tests it was decided to build cells into the small PPO 534 #281 cases (nominal 5 AH size). Since data was lacking on the performance of the smaller electrodes, the first experiment was designed to show differences between the electrodes that had been used in the past and those which would become standard for this phase of Task X support.

Twelve cells were constructed in Teflon inserts and then sealed in nickel bombs. The cells were sterilized at 135°C for 120 hours. The purpose of this test was to standardize the construction changes involved in the transition from 7-plate cells employing S-7.5 plates to 13-plate cells employing #281 plates. Six of these cells contained seven #281 plates and six contained thirteen #281 plates. The data are shown in Table X. The capacities per gram of silver were not as good as those obtained for seven-plate cells previously reported. The lower capacities of the recent cells were probably due to the way the negative electrodes were prepared. In earlier studies, the zinc mix was placed in a die lined with EM476. After pressing, the EM476 wrapper was removed and the electrode was wrapped in SWRI-GX retainer. However, the recent electrodes were pressed directly into the SWRI-GX retainers. When the latter process is used, a significant variation in electrode thickness occurs. These results indicate that some attention to pressing the zinc electrode is needed.

Following the above experiment, four cells were assembled, activated, sealed and sterilized in PPO 534 #281 cases. The purpose was to generate a procedure capable of producing a successful "400 cycle" cell employing existing equipment, tools, and techniques. All four cells leaked during

TABLE X

Capacity, Voltage, and Pressure of Seven and Thirteen-Plate Cells Sealed, Then Sterilized

	7-Plate Cells							13-Plate Cells				
	46	47	48	49	50	51	52	53	54	55	56	57
Cycle #1												
AH/g Ag	.34	.31	.35	.33	.32	.34	.33	.32	.34	.34	.33	.33
Midvoltage	1.42	1.43	1.42	1.42	1.42	1.43	1.38	1.38	1.38	1.38	1.38	1.38
Pressure Change at End of Cycle (psi)	none	none	none	-.9	-.9	-.5	+.6	+.4	+.7	+.6	+.7	+.5
Cycle #2												
AH/g Ag	.30	.27	.29	.28	.30	.30	.32	.31	.32	.32	.33	.31
Midvoltage	1.43	1.45	1.43	1.45	1.43	1.40	1.40	1.41	1.41	1.41	1.41	1.41
Pressure Change at End of Cycle	+.3	none	none	none	none	none	+.5	none	none	none	none	none
Cycle #3												
AH/g Ag	.28	.27	.27	.27	.27	.27	.31	.31	.32	.30	.30	.30
Midvoltage	1.44	1.44	1.44	1.44	1.44	1.44	1.43	1.42	1.42	1.42	1.42	1.41
Pressure Change at End of Cycle	none	none	none	none	none	none	none	none	none	none	none	none

(continued)

TABLE X (continued)
Capacity, Voltage, and Pressure of Seven and Thirteen-Plate Cells Sealed, Then Sterilized

	7-Plate Cells						13-Plate Cells					
	46	47	48	49	50	51	52	53	54	55	56	57
Cycle #4												
AH/g Ag	.29	.28	.28	.28	.28	.28	.30	.29	.29	.28	.30	.32
Midvoltage	1.41	1.41	1.41	1.41	1.41	1.41	1.39	1.39	1.39	1.39	1.39	1.39
Pressure Change at End of Cycle	+.7	+2.0	+2.5	+2.3	+2.0	+3.0	+.3	+1.4	none	+.3	-.5	none
Cycle #5												
AH/g Ag	.30	.28	.28	.28	.28	.28	.28	.28	.30	.29	.29	.31
Midvoltage	1.41	1.41	1.41	1.41	1.41	1.41	1.39	1.39	1.39	1.39	1.39	1.39
Pressure Change at End of Cycle	-1.2	-2.2	-3.5	-2.3	-2.9	-3.7	-.6	-1.6	none	-.6	none	none
Cycle #6												
AH/g Ag	.30	.29	.28	.28	.28	.27	.27	.28	.30	.29	.30	-
Midvoltage	1.42	1.42	1.42	1.42	1.42	1.42	1.39	1.39	1.39	1.39	1.39	-
Pressure Change at End of Cycle	none	none	none	none	none	none	none	none	none	none	none	-

sterilization, and this fact points up the need for an adequate sealing technique. The cells will be cycled mainly to determine whether any problems may be encountered in using the new cycling equipment that we intend to employ in further work.

FABRICATION AND TESTING OF CELLS

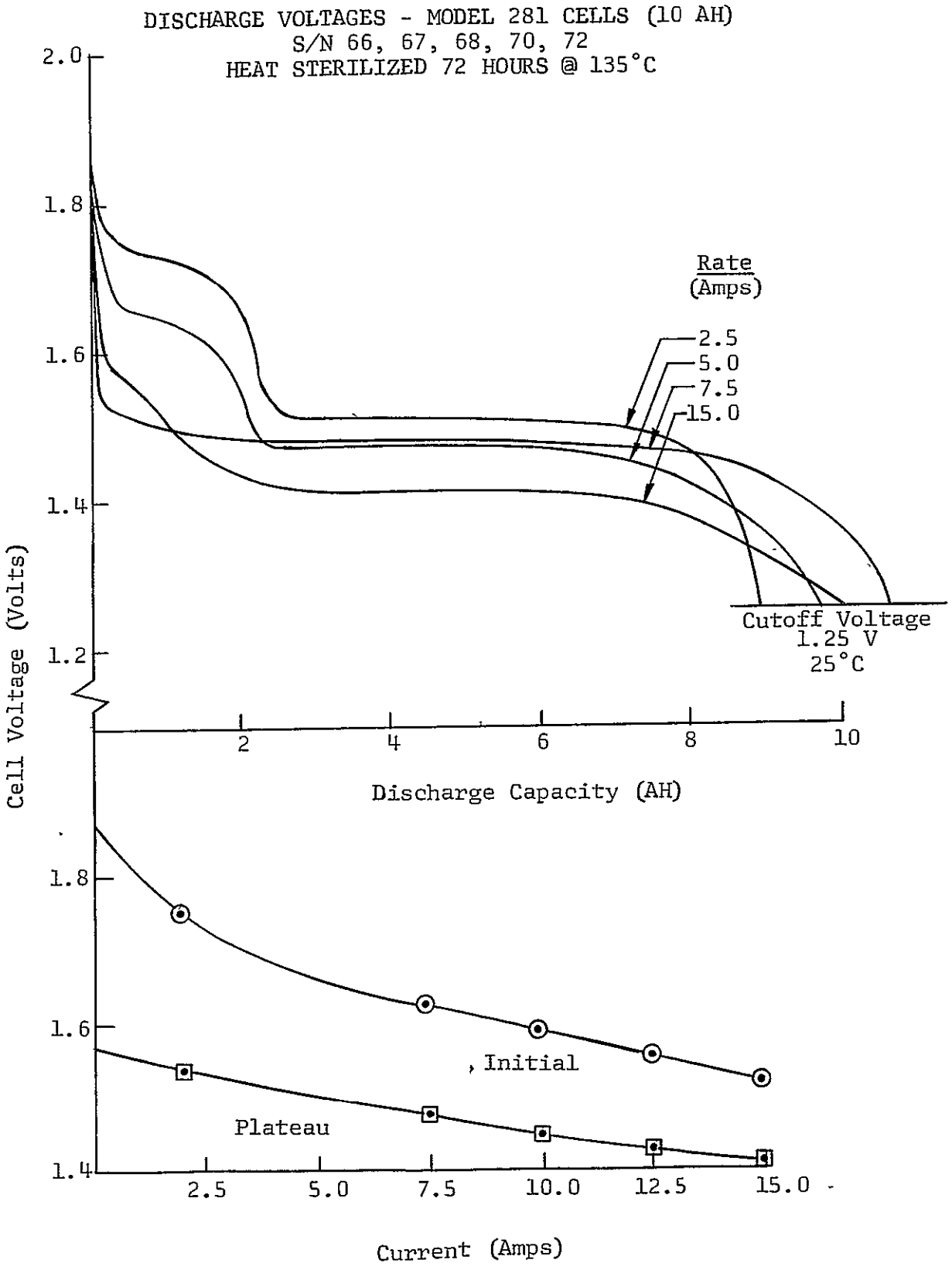
F. DEVELOPMENT OF HEAT STERILIZABLE - HIGH IMPACT 5.0 AH CELLS, TASK 9

A. Objectives and Past Work. - This task requires the development of cells meeting the requirements of JPL Specification GMP-50437-DSN-C, high impact of 2,800 ± 200 g at 115 ± 3 ft/sec, and JPL Engineering Memorandum 342-70. Thirty (30) cells of 5 AH nominal capacity are to be delivered in December 1969. Model 281 non-high impact test cells have delivered 11.0 AH at 3.3 amperes to 1.25 volts per cell, 16.4 watt-hours at an average voltage of 1.49 volts, and 43 watt-hours per pound of cell after 72 hours wet heat sterilization at 135°C. One high impact version of this cell constructed in the same cell case was assembled into a 12-cell battery and survived tests in the C-SAD experimental spacecraft after heat sterilization[1] at a shock level estimated to be 2400 "g".

B. Development Testing of Model 281 Cells. - Cycling of three 10 AH Model 281 test cells has continued at discharge rates of 0.25C, 0.50C, 0.75C, and 1.5C. Figure 1 summarizes voltage regulation and discharge capacities to 1.25 volts per cell observed during cycles 2 to 6 after 72 hours heat sterilization at 135°C. This 3-cell group (S/N 66, 67, and 68) was assembled, activated, and sealed with optimum processing and without heat shrinkable polyolefin tubing lead protection. No gassing during formation charge was noted and performance to date has been excellent.

Epoxy bond strength in sealing operations has been improved by sand blasting mating surfaces. To check the effect of sand blasting on cell gassing and as a further proof of the effect of heat shrinkable

FIGURE 1


DISCHARGE VOLTAGES - MODEL 281 CELLS (10 AH)
S/N 66, 67, 68, 70, 72
HEAT STERILIZED 72 HOURS @ 135°C
Cell Voltage (Volts)
2.0
1.8
1.6
1.4
1.2
Rate
(Amps)
2.5
5.0
7.5
15.0
Cutoff Voltage
1.25 V
25°C
2
4
6
8
10
Discharge Capacity (AH)
1.8
1.6
1.4
Initial
Plateau
2.5
5.0
7.5
10.0
12.5
15.0
Current (Amps)

polyolefin tubing (Raychem RNF-100), three more cells were assembled with optimum processing and the variables:

S/N 70 - Sand blasted container, no RNF-100 tubing

S/N 71 - RNF-100 tubing on plate leads, no sand blasting

S/N 72 - Control - No sand blasting, no RNF-100 tubing

Formation charge acceptance after heat sterilization for 72 hours at 135°C was 11.1, 11.2, and 10.7 AH respectively; however cell S/N 71 with RNF-100 bulged 120 mils from cell gassing, was vented, and later dissected. Table XIsummarizes the capacity data from the first four cycles on the non-gassing cells showing no major sand blasting effect.

In order to withstand the environmental forces during vibration at lift-off and soft landing and the deceleration "g" forces at soft landing, an epoxy platelock at the bottom of the cell jar is being considered. Cell S/N 73 was made identical to cell S/N 72 except that 2 cc of epoxy was added on the bottom of the plates to cement the "U" folds and exposed plates to the jar bottom. Table XI gives the first three cycles after 72 hours wet heat sterilization at 135°C showing discharge efficiencies in the same range as the cells without platelocks.

High impact cell designs will be dependent upon plates with high tensile-buckling strength cores. Positives are to be supported by .010" zirconium sheet sheathed with sintered DP active silver. Cells S/N 74 and 75 having 20% less silver active material (by weight) were assembled and tested in the Model 281 cell jar. All other design parameters were the same as S/N 72. Table XI gives two cycles of data after 72 hours

TABLE XI

CYCLING CAPACITIES OF MODEL 281 TEST CELLS

Test	Unit	Non-High Impact Design			High Impact Positives	
		S/N 70 SB, NP(4)	S/N 72 NSB, NP	S/N 73 NSB, P(3)	S/N 74 NP, Z	S/N 75 NP, Z
Cycle 1 (Formation)						
o Charge	AH	11.1 (1)	10.7 (1)	11.4 (1)	7.6	7.5 (1)
o Discharge (3.3/.7A)	AH	10.6	9.9	10.3	7.0	6.5
E (Efficiency)	AH/gm Ag	0.36	0.34	0.35	0.30	0.28
Cycle 2						
o Charge	AH	9.4	8.8	10.3 (1)	7.1	7.1 (1)
o Discharge (3.3/.7A)	AH	8.9	8.4	10.6	6.1	6.2
E	AH/gm Ag	0.30	0.29	0.36	0.26	0.26
Cycle 3						
o Charge	AH	10.2 (1)	10.2 (1)	9.9		
o Discharge (7.5A)	AH	9.9	9.3	9.5		
E	AH/gm Ag	0.33	0.32	0.32		
Cycle 4						
o Charge	AH	8.5	8.2	--		
o Discharge (5.0A)	AH	8.7	8.2	--		
E	AH/gm Ag	0.30	0.28	--		
Bulge, max. in cycling	mils	3	3	7	1	0

NOTES:

(1) Charge included a partial discharge recharge.

(2) S/N 70, 72, 73 have 29.4 gm active Ag; S/N 74, 75 have 23.4 gm Ag as positive plate core.

(3) S/N 73 identical to S/N 72 except has 2 cc Epocast 221/927 platelock.

(4) SB = sandblasted sealing areas
NSB = no SB
P = platelock
NP = no P
Z = Zirconium core in positive plate.

heat sterilization at 135°C. Discharge efficiencies are lower by 17-21%. Figure 2 shows reference electrode and cell voltages for cycle 3 charge. Figure 3 gives reference electrode and cell voltages for cycle 3 discharge. In both cases the positive Zr reinforced plate limited performance.

C. Effect of Heat Shrinkable Polyolefin Tubing on Negative Plate Gassing. - An experiment devised to show the effect of the presence of various insulating tubing on the gassing rate of zinc negatives consisted of placing a dry charged negative plate in intimate contact with lengths of tubing while submerged in 40 weight percent KOH saturated with zinc oxide. Gas was collected for 48 hours at 140°F from test assemblies including:

- Only zinc plate.
- Zinc plate plus RNF-100 heat shrinkable polyolefin tubing.
- Zinc plate plus teflon tubing, non-heat shrinkable.
- Zinc plate plus teflon tubing, heat shrinkable.

Zinc area and tubing length was controlled in each test. In all cases hydrogen collected from negatives exposed with RNF-100 tubing was 2-3 times greater than from the other test conditions. Figure 4 shows typical gassing curves, RNF-100 assemblies exhibiting a constant 0.83 cc/hr. gassing rate while zinc alone and zinc with all other tubing tested peaked out at 10 to 20 cc in 2 days exposure at 140°F. No gas was evolved from RNF-100 tubing in the absence of zinc. No significant differences in gassing rates were noted between zinc alone and zinc plus the teflon tubing selected for use in Task 13 cell designs.

FIGURE 2

CHARGE VOLTAGES - MODEL 281 CELL
WITH ZR - REINFORCED POSITIVES

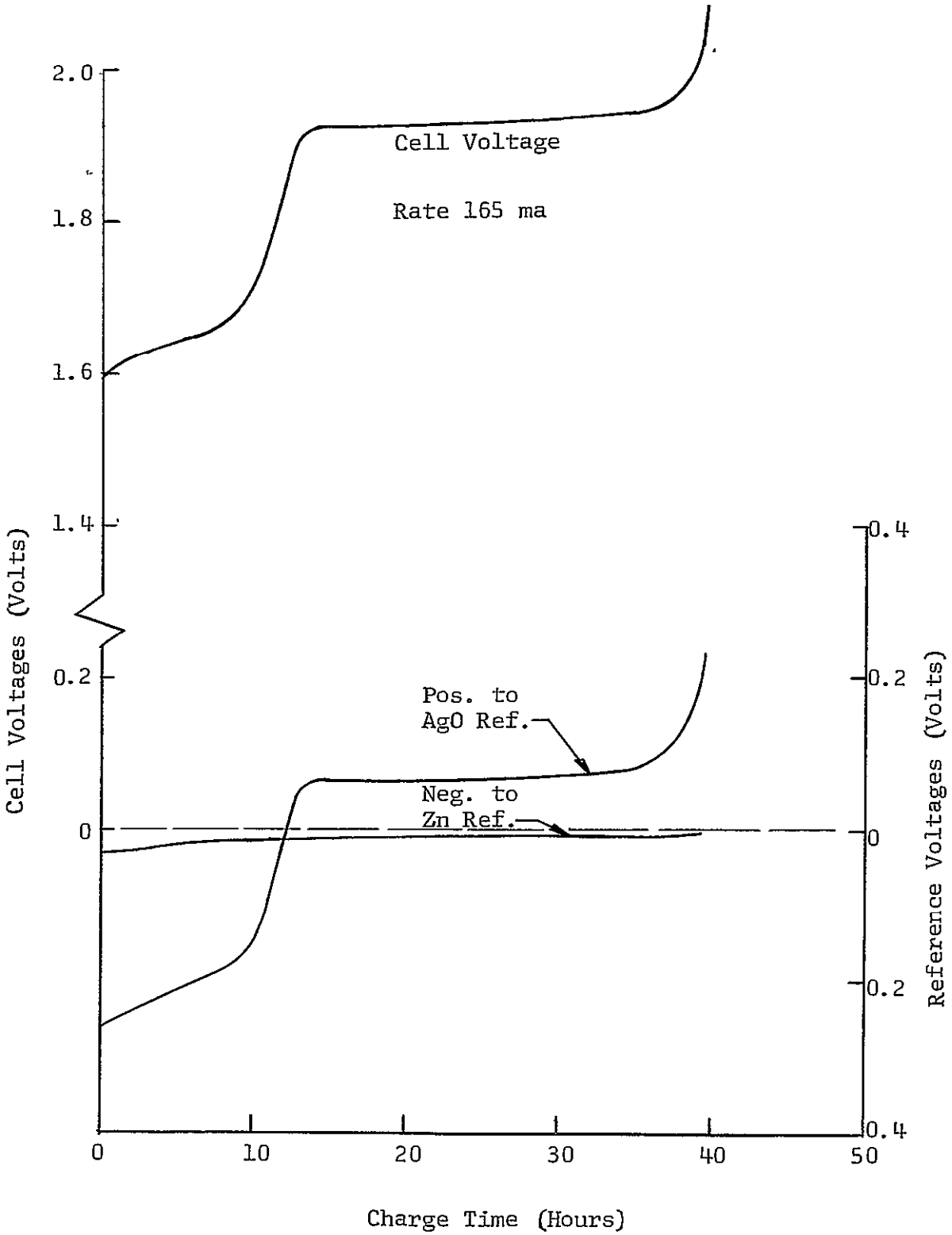


FIGURE 3

DISCHARGE VOLTAGES-MODEL 281 CELLS WITH
ZR - REINFORCED POSITIVES

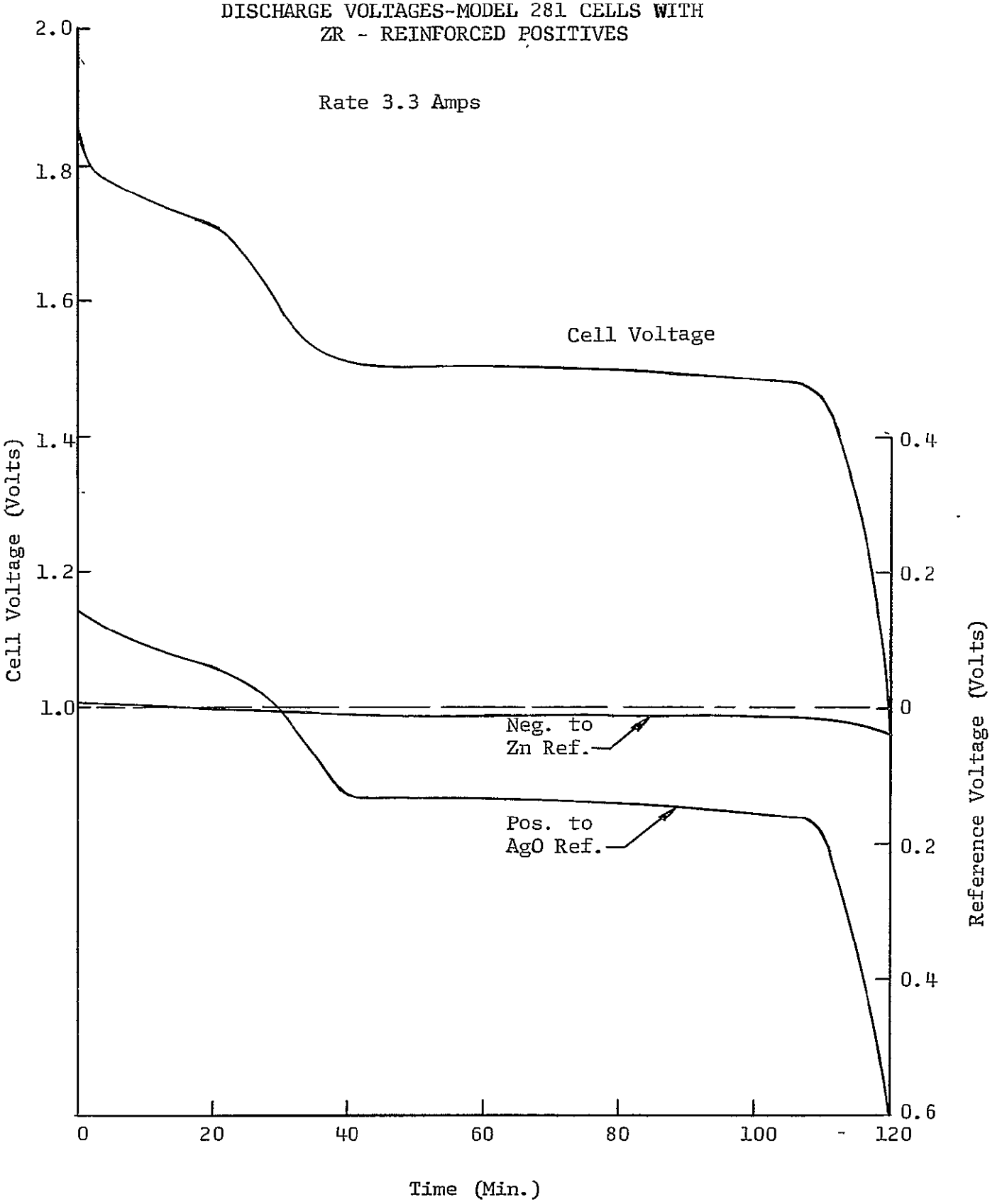
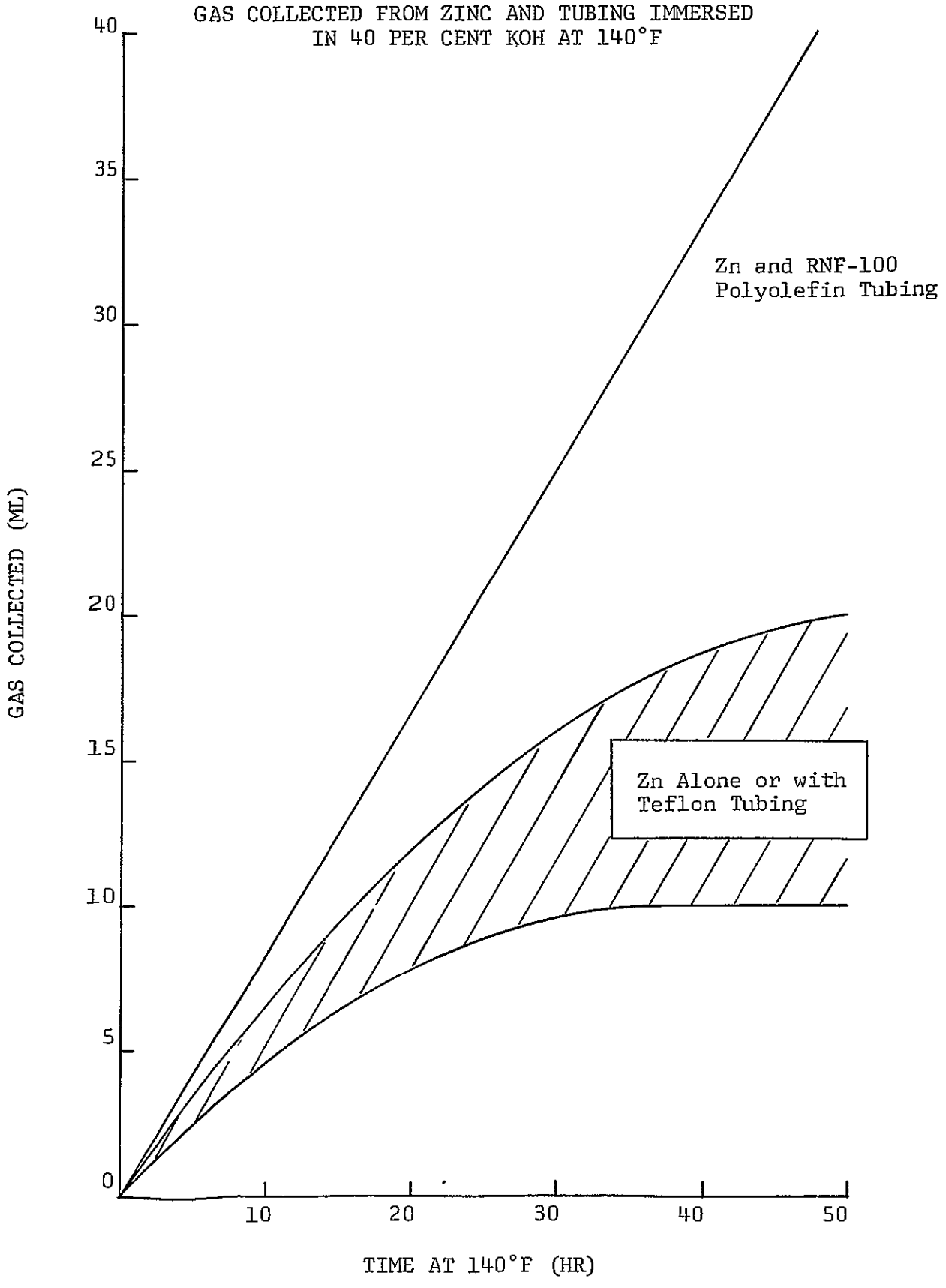


FIGURE 4


GAS COLLECTED FROM ZINC AND TUBING IMMERSED
IN 40 PER CENT KOH AT 140°F
GAS COLLECTED (ML)
0
5
10
15
20
25
30
35
40
10
20
30
40
50
TIME AT 140°F (HR)
Zn and RNF-100
Polyolefin Tubing
Zn Alone or with
Teflon Tubing

D. Model 361 5.0 AH High Impact Cell Design. - Design of the 3,000 "g" 5.0 AH cell has been completed through prototype design review. Molded PPO cell case and cover parts are being procured for prototype cell tests during the next quarter.

Cell design features for the 5.0 AH and 25 AH cell are alike except for the number of plates in each cell. Plate design and processing are described in Task 12.

E. Future Work. - During the next quarter tests to improve zirconium positive efficiencies will continue. Etched Ag grids for high impact negatives will be procured in sufficient quantity to construct four 5.0 AH cells in Lucite jars for design proofing tests at 2,000, 3,000 and 4,000 g's at JPL and to determine electrochemical performance.

II. DEVELOPMENT OF HIGH CYCLE LIFE 48 AH CELLS TASK 10

A. Objectives and Past Work. - This task requires development of wet heat sterilizable 48 AH cells meeting the requirements of JPL Specification 50436-DSN-B and delivery of 100 cells in April 1970. Operational requirements include:

- One year prelaunch storage followed by 72 hours heat sterilization at 135°C;
- Charge, 9-month interplanetary travel;
- Environmental shock 200 "g" for 0.7 ± 0.2 msec, 100 "g" deceleration for 10 minutes, and 35 "g" rms maximum vibration 100 to 2000 cps; plus after landing;
- 400 cycles, 50% depth, 12-hour maximum charge time and discharge rates of C/10 to C/3 from 10°C to 50°C.

Half size 24 AH cells have shown failure by Ag and Zn penetration which has limited life to 84 cycles in 5-6 months wet life for 6 layers of Southwest Research Institute GX separator system. Erosion of dry-teflonated unsintered negatives reduced capacity at a rate of 0.33 AH/cycle.

B. Factorial Experiment. - Twenty-seven 16 AH cells have been designed and assembled according to the factorial design given in Table XII. Each cell has in common 4 full negative plates wrapped in 9 layers GX separator and five positive plates (3 full and 2 half thickness). Polyphenylene oxide shims make up the difference between cell jar inside length and pack thickness for each of the 27 designs. All negative plates contain 4 weight percent compound 323-43, a grid assembly of two 3/0 expanded silver grids welded together, and an active material density of 45 gm/in^3 after sintering.

Cell design equations derived for the factorial conditions are listed below:

$$T^- = \frac{W\,T^+\,d^+}{f^-\,d^-} = \frac{(.029\text{ in.})(69\text{ gm/in}^3)}{(45\text{ gm/in}^3)} \cdot \left(\frac{W}{f^-}\right) \qquad \text{Equ. 1}$$

$$S = 0.583 - 0.178\left(\frac{W}{f}\right) - 72\,T_m \qquad \text{Equ. 2}$$

where T = thickness active material only

W = zinc oxide to silver weight ratio

d = Apparent active material density

f = % zinc oxide in negative mix

S = Shim thickness

T_m = Wet thickness of membrane

TABLE XII

CELL DESIGN VARIABLES AND ASSIGNED COMBINATIONS
FOR CYCLE LIFE STUDY

CELL DESIGN VARIABLES

Variable	Factor Code Level		
	-1	0	1
A. Weight percent Teflon 7	5	7	9
B. Separator Wet Thickness Allowance (mils)	2.0	2.4	2.8
C. Zinc Oxide to Silver Weight Ratio	.9	1.2	1.5
D. Electrolyte Concentration Before Saturation (%)	41	43	45

VARIABLE COMBINATIONS

Cell No.	Variable and Level				Cell No.	Variable and Level			
	A	B	C	D		A	B	C	D
1	-1	-1	-1	-1	15	1	1	0	0
2	0	1	0	1	16	-1	0	0	0
3	1	0	1	0	17	0	-1	1	-1
4	-1	-1	1	0	18	1	1	-1	1
5	0	1	-1	-1	19	-1	1	-1	0
6	1	0	0	1	20	0	0	0	-1
7	-1	-1	0	1	21	1	-1	1	1
8	0	1	1	0	22	-1	1	1	1
9	1	0	-1	-1	23	0	0	-1	0
10	-1	0	-1	1	24	1	-1	0	-1
11	0	-1	0	0	25	-1	1	0	-1
12	1	1	1	-1	26	0	0	1	1
13	-1	0	1	-1	27	1	-1	-1	0
14	0	-1	-1	1					

During plate manufacture two problems were encountered and resolved. Half positives made by pressing one sheet of DP silver into the Ag grid left grid exposed. New half positives were made by pressing one sheet of half thickness DP silver onto each side of the grid. Negative plates were found to crumble along plate edges where grid tolerances were on the low side and did not fully cover the pressing cavity. The mean weights of negative active material for the 27 cells (nine negative plate designs) are on the low side of the tolerance range. On future cells grid size and plate size will be identical in overall width and height.

Procurement problems were encountered and resolved. Molded polyphenylene oxide jar covers exhibited sink deformation in heavy cross-sections. The dimensions were out of tolerance because of this shrinkage in the critical seal area. Heavy cross sections were reduced by adding ten pins in the mold. Parts now on hand are to print and have passed sealing tests.

The 27 cells will be activated and heat sterilized prior to initiation of 100% depth constant current cycling tests during next quarter. Two charge-discharge cyclers are on order from Electro-Mechanical Research, Greenbelt, Maryland.

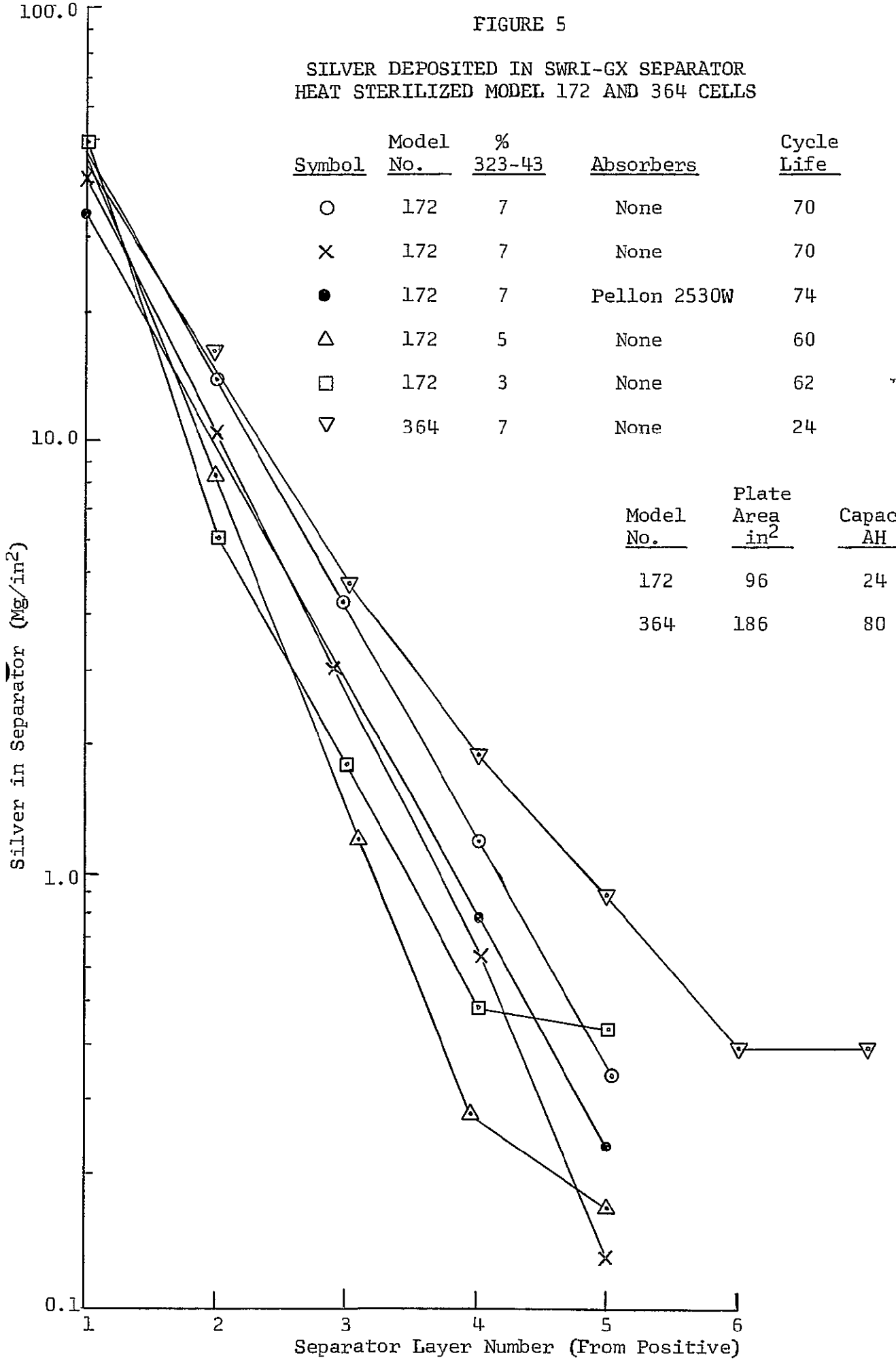
C. Model 172 Cell Silver Analyses. - Preliminary cycling tests on 15 Model 172 cells and cycle life to failure were reported previously [2]. Heat sterilized cells failed by Ag and Zn penetration while non-sterile control cells gave 30-40% more cycles and failed by zinc erosion. Analyses of the separator membranes of a single cell from each test group yielded the results of Figure 5. A semi-log plot of silver in

FIGURE 5

SILVER DEPOSITED IN SWRI-GX SEPARATOR
HEAT STERILIZED MODEL 172 AND 364 CELLS

Symbol	Model No.	% 323-43	Absorbers	Cycle Life	Wet Life
○	172	7	None	70	6.1
×	172	7	None	70	6.1
●	172	7	Pellon 2530W	74	6.1
△	172	5	None	60	5.5
□	172	3	None	62	5.5
▽	364	7	None	24	10.0

Model No.	Plate Area in^2	Capacity AH
172	96	24
364	186	80



mg/in^2 for each layer, positive plate to negative plate, is linear with wet thickness. Degree of deposition does not appear to be related to the weight percent of compound 323-43 or wet life but rather to the total accumulated discharge capacity:

Cell S/N	% 323-43	Total Cycles 100-80-60% DOD	Wet Life Months	Discharge Capacity (AH)	Deposited Ag mg/in^2 3rd	4th
4, 5	7	70	6.1	1245	3.6	0.9
12	5	60	5.5	1117	1.2	0.3
15	3	62	5.5	1158	1.8	0.5

Total cell life in terms of accumulative discharge capacity for cells S/N 4 and 5 after 72 hours heat sterilization was 1245 AH/96.6 gms Ag/5L GX membrane. Shorting occurs when Ag deposits in the layer adjacent the negative plate increase to 0.1 to 0.5 mg/in^2.

D. Future Work. - During the next quarter the factorial experiment cells will be activated and sealed, heat sterilized, and then cycled at 100% depth of discharge at constant current to failure to deliver 50% of rated capacity.

III. DEVELOPMENT OF RECHARGEABLE PRIMARY 70 AH CELLS TASK 11

A. Objectives and Past Work. - In this task wet heat sterilizable 70 AH cells are being developed to meet the requirements of JPL Engineering Memorandum 342-71 summarized previously[3]. One-hundred cells are presently scheduled for delivery in late August 1969. The Model 364 cell has demonstrated to date mean 4 cycle discharge capacity of 89 AH at the C/4 rate, 133 watt-hours at an average voltage of 1.49 volts, and an average density of 52 WH/lb. of cell. Cell life to date is 14 months.

B. Cell Redesign and Plate Fatigue Tests. - Model 364 cells failed during vibration testing by positive plates buckling and breaking below the plate lugs. One cell, S/N 2, dissected after 10 months and 24 cycles of life, revealed the additional problem areas -

- Negative plate erosion as high as 44%.
- SWRI-GX six layer separator system and GX retainer firmly adhered, separable only after soaking in water.
- Negative plate grids extremely friable.

Cell grid structures have been redesigned and evaluated by means of a plate fatigue tester designed to simulate vibrational tension forces on plates at varying frequencies and "g" loads. Negative plate erosion has been attacked by changing to a sintered teflonated plate process. Amalgamation has been reduced 50% to reduce loss of grid strength and grids have been framed by a pure silver channel. Separator adhesion has been reduced by heat treatment of the cell pack in the flooded state at 100°C.

Fatigue test criteria were based on the assumptions that greatest cell pack movement usually occurs at 100 to 200 Hz, a JPL test procedure requiring 22 minutes to complete the sweep of 2 to 2,000 to 2 Hz, and a constant 1950 cycles per minute operation of the test fixture. The minimum acceptable time of test was calculated to be 135 minutes. Table XIII summarizes fatigue test results for 3 positive and 5 negative plate or grid assemblies leading to selection of the prototype plate designs.

TABLE XIII

70 AH CELL PLATE FATIGUE TESTS

Plate or Grid Type	Pattern of Grid	Weight gm/in^2	Other Feature	Amplitude ± Inches	Run Time To Failure Minutes	Failure Mode Or Comments
1. Previous Model 364 Positive Plate (formed)	10 Ag 15 2/0	.45	Edges Coined	0.06	1.3 86.	Below lug at solid to mesh interface
2. " "	"	"	Ag channel around plate edges	0.06 0.12	170. 27.	No damage. Lug severed at lug to plate interface
3. New Model 364 Positive Plate (formed)	3/0 2 Ply	.22	Ag channel around plate edges	0.06	217.	No damage. Recommended for cell tests
4. Negative Plate Uniformed, sintered	10 Ag 15 2/0	.45	.25" grid fold over Solid .010" Ag lug spotwelded 1/2 width plate	0.06	190.	60% active material lost at 30 seconds. No further damage.
5. " "	3/0 2 Ply	.22	" "	0.06	210.	Slight loss active material. No other damage.
6. " "	"	"	Same as above but no fold over	0.06 0.09	186. 28.	No damage. Bottom frame to grid interface
7. Negative Plate Formed, sintered	"	"	" "	0.06	156.	Slight loss zinc below lug. Recommended design.
8. Negative Grid only (Prototype design)	"	"	" "	0.06 0.09 0.12	420. 93. 23-30	No damage. At lug radius. At lug to side "U" frame.

C. Silver Analysis Model 364 Test Cell. - Development cell S/N 2 was dissected after a 10 month wet life and 24 charge-discharge cycles to 100% depth. On the last discharge reference electrodes verified negative plate limitation. No indication of shorting was observed at any time up to dissection. The separator system was removed and analyzed chemically for silver deposited on each layer of GX. Figure 5 summarizes the analytical results. Cell S/N 2 had a total accumulative discharge capacity of 1580 AH during 10.0 months wet life and 24 cycles. Comparison of the silver deposited on the 4th and 5th layers of the Model 364 cell with the same values from Figure 5 for the Model 172 cells leads to a cell design normalization based on the parameter mg Ag/in^2/AH discharge/month of wet life.

Model No.	Sample	Silver Deposit 4th L mg/in^2	5th L mg/in^2	Cycles	Accumulated Capacity AH	Wet Life Months	Normalization Factor mg/in^2/AH/mon. X 10^4
172	1	1.2	0.3				
	2	0.6	0.1				
	$\bar{X}_4$	.9		70	1245	6.1	1.2
	$\bar{X}_5$		0.2		--	--	0.3
364	1	1.9		24	1580	10.0	1.2
			0.9		--	--	0.6

This hypothesis should be tested by data from all heat sterilized cells having the same type GX separator to provide a better understanding of how cells fail by silver penetration.

D. Future Work. - During the coming quarter work scheduled includes:

- Design review of revised Model 364 cell.
- Manufacture nine prototype cells
- Burst pressure tests of jar and molded PPO covers.

IV. DEVELOPMENT OF HEAT STERILIZABLE HIGH IMPACT 25 AH CELLS TASK 12

A. Objectives and Past Work. - This task requires the development of wet heat sterilizable 25 AH cells capable of surviving 4,000 "g" shocks in any axis and meeting the requirements of JPL Engineering Memorandum 342-68 detailed previously[4]. A lot of 100 cells is to be delivered in December 1969.

B. Plate and Cell Jar Structures. - Previous work discussed in Task 9 resulted in the positive and negative plate designs of Figures 6 and 7. The positive plate, reinforced with .017" zirconium sheet, will hang within the "U" fold of 8L SWRI-GX membrane and be supported only by the 0.36 x 1.0 x .017 zirconium strut. This plate is more vulnerable to damage and is the lighter plate. The heavier zinc plate is supported by a massive Ag grid, chemically etched from pure Ag sheet, and the pasted assembly slides into slots in the PPO cell jar. Grid weight will be 13.3 ± 1.3 grams based on variability of the first lot received from National Engraving Service. Negative active material densities achieved to date are below design values requiring process improvement. Positive plates have been made successfully to print requirements. Slotted jars for holding the 19 negatives and 18 positives subcovers, spacers, and top covers have all been released for molding in PPO 534-801.

C. Test Cells for High Impact Design Verification. - Four experimental nine-plate test cells were activated and are scheduled for 2,000, 3,000, and 4,000 "g" impact at JPL. Motion pictures will be made of plate deflections under these loads during shock.

FIGURE 6

HIGH SHOCK POSITIVE PLATE

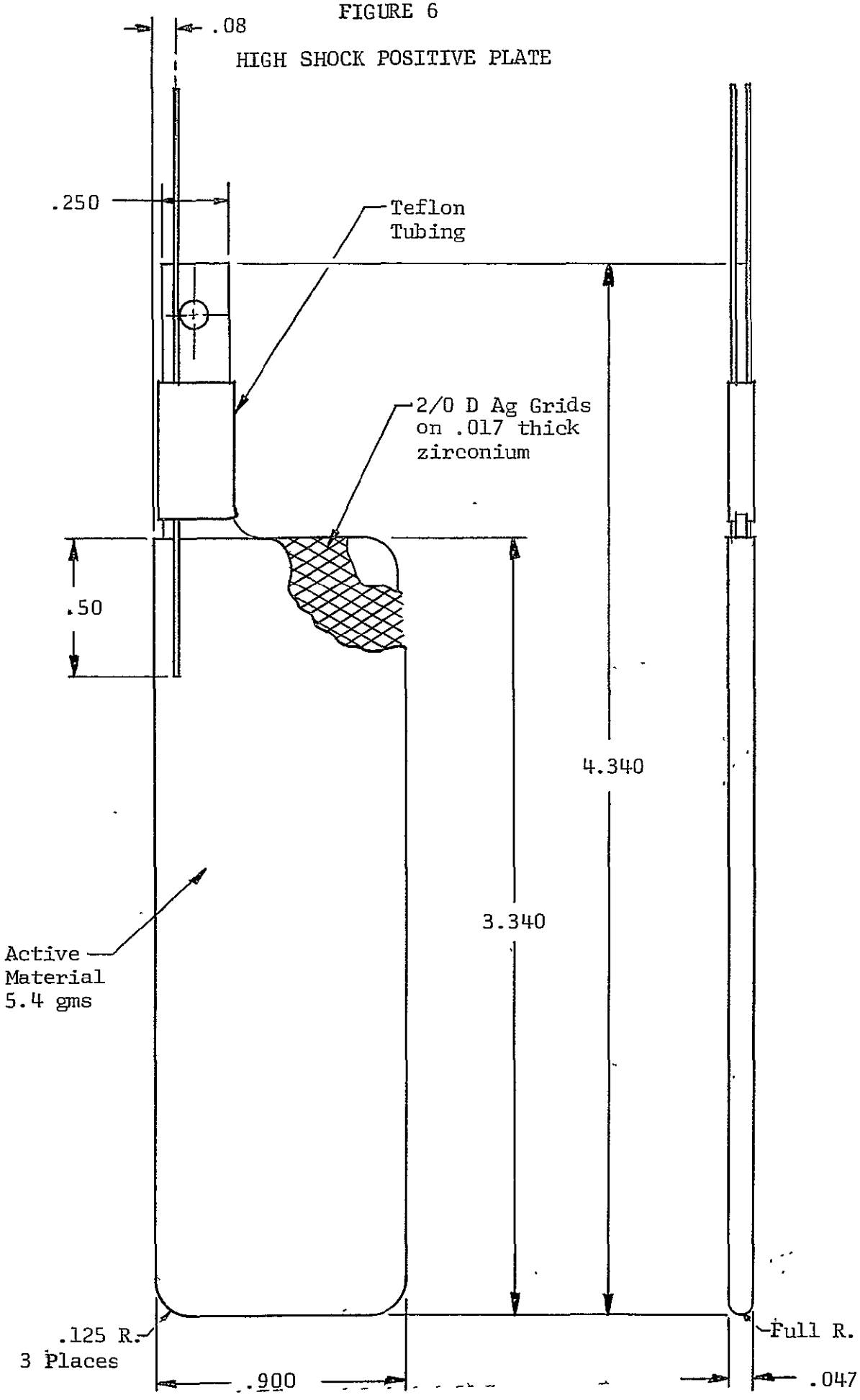
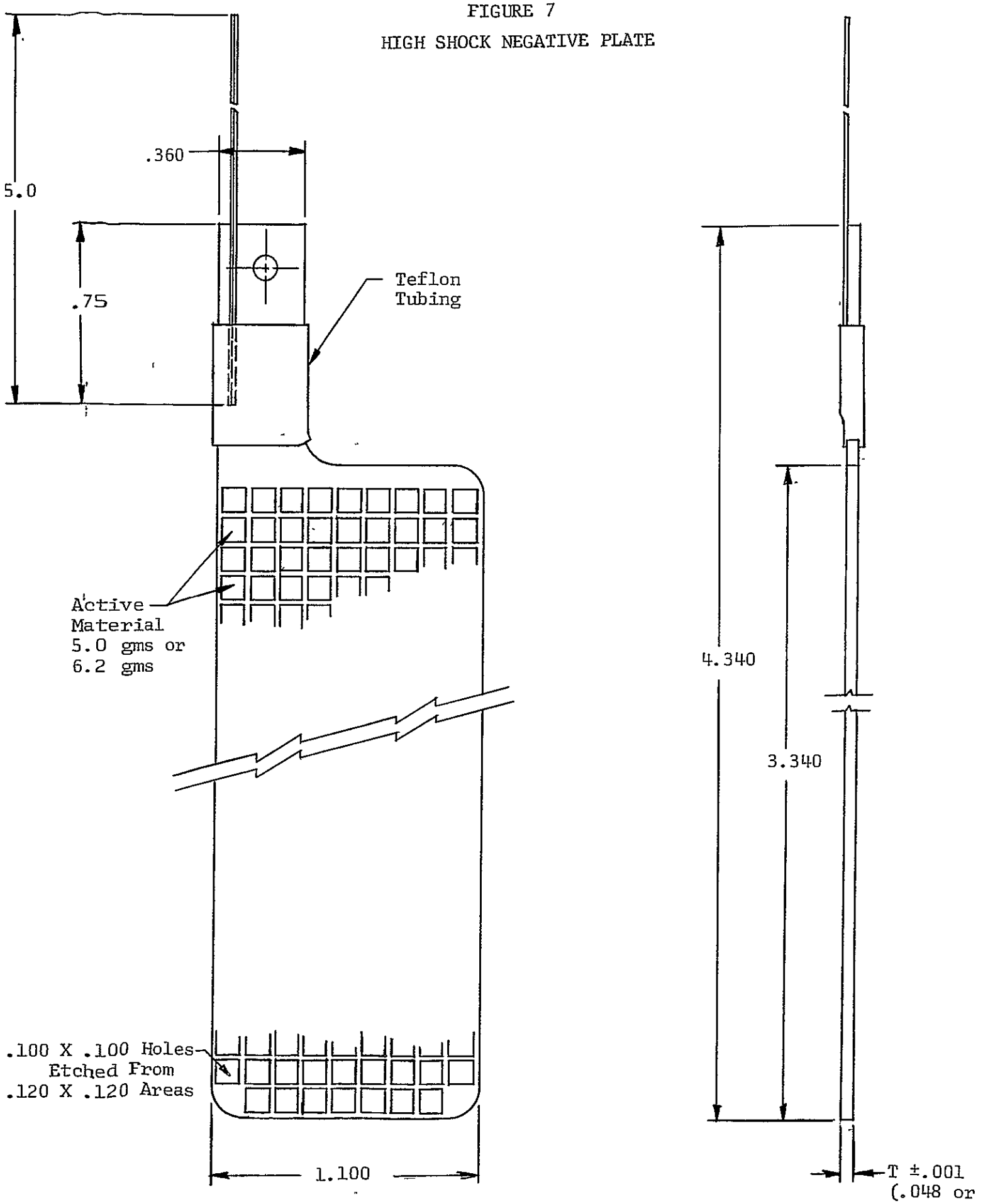


FIGURE 7

HIGH SHOCK NEGATIVE PLATE



D. Future Work. - During the coming quarter work will include:

- Design review of prototype Model 362 25 AH 4,000 "g" impact cell.
- Impact tests of non-sterile 9-plate cells at JPL.
- Release tools for assembly of cells.
- Cycling tests on surviving 9-plate cells.

V. DEVELOPMENT OF HEAT STERILIZABLE MEDIUM CYCLE LIFE 25 AH CELLS TASK 13

A. Objectives and Past Work. - In this task a 25 AH wet heat sterilizable cell will be designed, developed, and tested to the requirements of JPL Engineering Memorandum 342-68 (5) less the 4,000 "g" shock. ESB Model 379 cell has been designed in five versions and 25 cells released for production, 5 of each design type, for cycling tests to demonstrate capability for 90 50% depth of discharge cycles. Deliverable items include:

20 Development A cells	3/69
20 Development B cells	4/69
20 Development C cells	5/69
100 Final Design by	8/69

B. Model 379 Cell Designs and Formation Cycle Data. - Since the Model 372 25 AH cell had demonstrated a life to short of 60-84 cycles at 50% depth in 6 months, redesign was concentrated on the objectives:

- Increase cycle life to 90 50% DOD cycles reliably.
- Increase wet life from 6.0 to 18 months.
- Improve vibration and shock capability.

Five 25 AH cell designs were completed to test the design parameters: two negative plate active material densities, 42 and 49 gm/in^3; 7 or 9

layers of SWRI-GX membrane as the separator; and positive or negative wrapped plates. Common to all cells were the design features:

- ZnO/Ag formation charge ratios increased 20% to 1.2:1.0.
- Twin 3/0 grids, spotwelded together with diamond patterns at right angles, in each negative plate.
- Reduction of compound 323-43 from 7 to 4% in negative active material.
- Sintered teflonated negative plates.

Each design group (-1 to -5) consisted of 5 cells. To one cell from each group was added an epoxy platelock for vibration resistance. This group is called the "Platelock Test Group." One more cell from each group was withdrawn to be given three pretest cycles before heat sterilization. This group is called the "Pretest Group." Table XIV summarizes cell weights, voltages, and pre-formation inputs for cell S/N 1 through 20 by design group. The preformation charge and bake-outs are manufacturing processes which optimize later performance after heat sterilization. Fifteen cells were then heat sterilized for 72 hours at 135°C and placed on formation charge. Table XV gives the formation charge capacity and formation discharge capacity of the 15 sterilized cells with the 5 non-sterilized cells for comparison. Table XVI summarizes the effect of heat sterilization by design group and averaged over all groups, a loss of discharge efficiency and energy density of 6 percent. Range of 25 AH cell energy densities on the C/3 rate discharge was 43.7 to 50.3 WH/lb, considered acceptable performance. Figures 8 through 13 are plots of voltage versus discharge capacity for the discharges on the sterilized cells. Figure 13 gives curves for the

TABLE XIV
CELL WEIGHTS, OPEN-CIRCUIT VOLTAGE, AND PRE-FORMATION INPUTS FOR MODEL 379 CELLS

Cell Group	Cell P/N	Design Factors: Plates Wrapped	Design Factors: Separation	Design Factors: Negative Mix Density	Cell Weight After Adjustment (g)	Electrolyte Weight (g)	O.C.V. Before 1st Bake	O.C.V. After 1st Bake	Preformation Input (Amp-hr)	O.C.V. Before 2nd Bake	O.C.V. After 2nd Bake
1	1	Positives	7 Layers	49 gms	489.3	122.7	-.02	-.031	.82	1.36	.78
	2	↓	SWRI-GX	Per cu. in.	487.9	123.5	-.02	-.039	.79	1.54	.82
	3		1 Absorber	↓	491.3	122.7	-.02	-.038	.77	1.52	.79
	4	↓	Pellon 2530W	↓	490.6	122.7	-.02	-.046	.87	1.54	.82
2	5	Positives	↓	42 g per	123.9	-.03	-.03	-.034	.70	1.50	.05
	6	↓		cu. in.	490.2	125.4	-.02	-.039	.72	1.54	.79
	7			↓	484.6	124.2	-.02	-.037	.64	1.38	.05
	8	↓	↓		483.0	124.7	-.02				
3	9	Positives	9 Layers		493.6	124.9	-.10	-.044	.81	1.37	.81
	10	↓	SWRI-GX		486.9	124.9	+.03	-.046	.80	1.56	.97
	11		↓		486.4	128.8	+.05	-.446	1.30	1.58	1.56
	12	↓			490.4	124.9	+.17	-.061	.80	1.55	.82
4	13	Negatives			488.0	123.7	+.28	-.039	.80	1.51	.78
	14	↓			489.1	123.3	+.29	-.043	.79	1.54	.84
	15				485.7	123.9	+.27	-.039	.74	1.55	.82
	16	↓		↓	490.4	123.8	+.27	-.040	.77	1.53	.83
5	17	Negatives	↓	49 g per	502.4	117.7	+.28	-.037	.75	1.36	.06
	18	↓		cu. in.	503.4	118.2	+.23	-.039	.72	1.54	.82
	19			↓	499.8	117.7	+.27	-.041	.74	1.38	.79
	20	↓	↓	↓	503.4	119.4	+.22	-.039	.69	1.54	.82

TABLE XV

FORMATION CHARGE AND DISCHARGE CAPACITIES OF MODEL 379 CELLS

Cell Group	Cell S/N	Design Factors: Separation	Design Factors: Negative Mix Density	Formation Charge (AH)	Partial Discharge (AH)	Net Input (AH)	Charge Efficiency (AH/g Ag)	Discharge Capacity (AH)	Discharge Efficiency (AH/g Ag)	Discharge Efficiency (% of Input)
1	1	Positive Wrap	49g/cu.in.	--	--	--	--	--	--	--
	2 (1)	7 Layers GX		30.2	4.9	34.5	.388	33.6	.379	97.4
	3	1 Pellon 2530W		30.0	7.0	34.3	.386	31.5	.354	91.8
	4	Absorber		31.5	5.6	34.2	.385	31.6	.355	92.3
2	5	↓	42g/cu.in.	30.2	7.5	33.6	.415	30.6	.378	91.1
	6			32.6	10.2	39.2	.484	35.4	.437	90.2
	7			30.2	9.8	37.4	.462	33.4	.412	89.3
	8 (1)			32.7	8.4	37.5	.463	36.0	.444	95.9
3	9	Positive Wrap	42g/cu.in.	34.3	4.0	36.2	.440	34.0	.413	93.8
	10	9 Layers		32.0	1.6	33.1	.402	30.6	.372	92.3
	11 (1)	GX		34.1	3.6	35.4	.430	34.4	.418	97.2
	12			32.4	4.4	34.7	.422	31.8	.387	91.6
4	13	Negative Wrap	42g/cu.in.	--	--	--	--	--	--	--
	14			33.9	7.1	39.6	.418	36.0	.380	90.8
	15 (1)	9 Layers		34.5	3.6	36.4	.384	36.6	.386	100.1
	16	GX		33.2	7.9	38.0	.401	35.6	.376	93.7
5	17	↓	49g/cu.in.	36.9	2.0	40.8	.394	37.1	.358	90.8
	18 (1)			42.7	5.3	44.3	.427	43.5	.419	97.9
	19			36.9	1.3	41.2	.397	37.2	.359	90.3
	20			35.8	7.2	41.1	.397	38.0	.367	92.3

(1) *Un-sterilized cells.*

TABLE XVI

MEAN AND RANGE OF CHARGE AND DISCHARGE CHARACTERISTICS FROM MODEL 379 CELL GROUPS

Cell Group	Formation Charge Efficiency (AH/g Ag)		Formation Discharge Efficiency (AH/g Ag)		Discharge Efficiency % of Net Input)		Discharge Voltage at 50% delivered Capacity		Energy Density (Watt-Hrs/Lb.)	
	Sterile	Non-Sterile (1)	Sterile	Non-Sterile (1)	Sterile	Non-Sterile (1)	Sterile	Non-Sterile (1)	Sterile	Non-Sterile (1)
1	.386 (.386-.385)	.388	.354 (.355-.354)	.379	92.0 (92.3-91.8)	97.4	1.50	1.48	43.7	46.2
2	.454 (.484-.415)	.463	.409 (.437-.378)	.444	90.2 (91.1-89.3)	95.9	1.50	1.48	46.1	49.3
3	.421 (.440-.402)	.430	.391 (.413-.372)	.418	92.6 (93.8-91.6)	97.2	1.50	1.47	44.7	46.8
4	.410 (.418-.401)	.384	.378 (.380-.376)	.386	92.2 (93.7-90.8)	100	1.49	1.49	49.5	50.4
5	.396 (.397-.394)	.427	.361 (.367-.358)	.419	91.9 (92.3-90.3)	97.9	1.50	1.48	50.3	57.7
All Groups	.416 (.484-.385)	.418 (.463-.384)	.379 (.437-.354)	.402 (.444-.379)	91.6 (93.8-89.3)	97.7 100-95.9	1.50	1.48	46.7	50.1
% Change Due to H.S.	-.5	0	-5.7	0	-6.2	0	+1.4	0	-6.8	0

(1) One cell per group.

FIGURE 8

DISCHARGE VOLTAGE VS CAPACITY, MODEL 379 NON-HEAT STERILIZED CELLS
8 AMPS TO 1.25 V AT ROOM TEMPERATURE

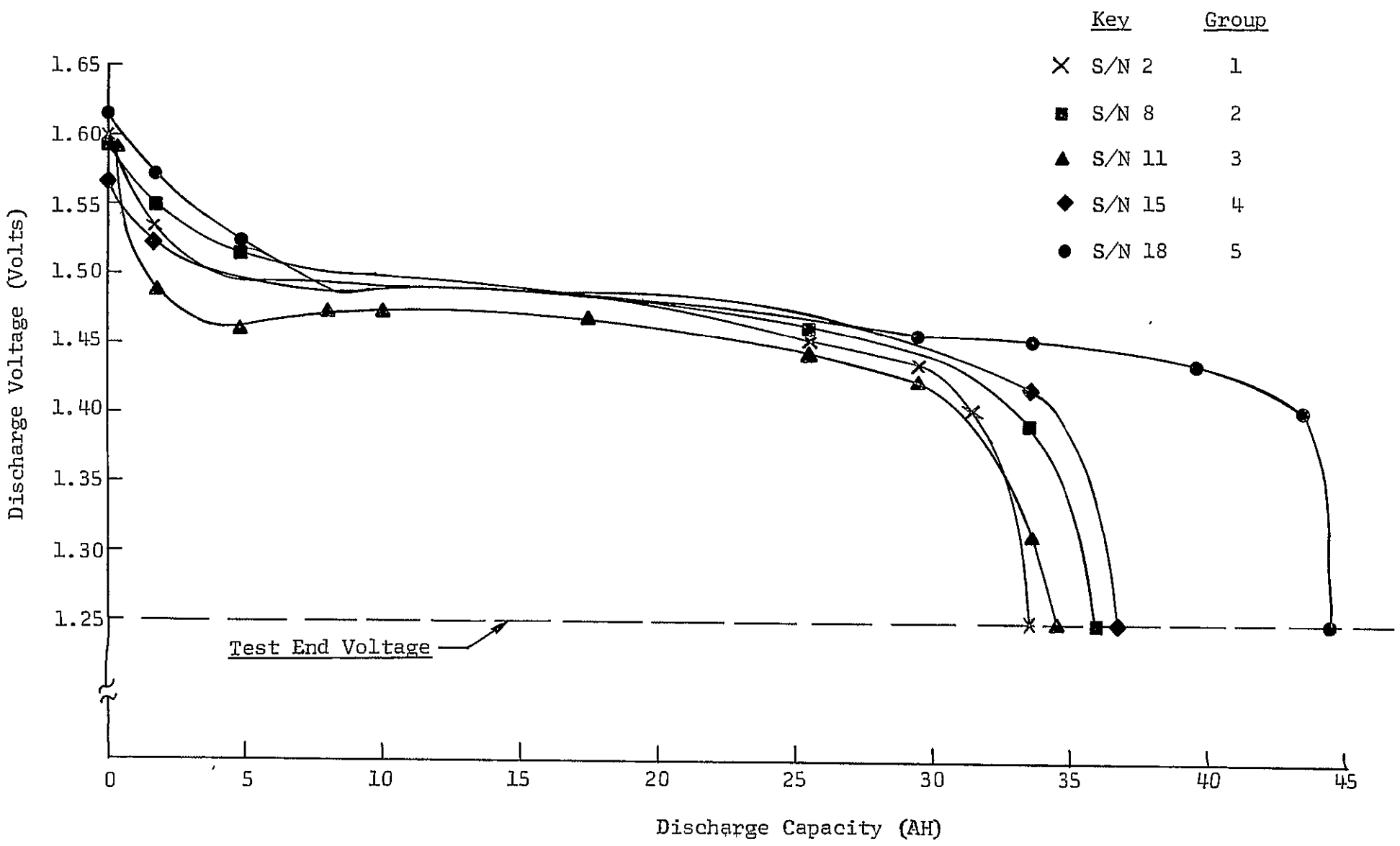


FIGURE 9

DISCHARGE VOLTAGE VS CAPACITY, MODEL 379
TYPE 1 HEAT STERILIZED CELLS
8 AMPS TO 1.25 V AT ROOM TEMPERATURE

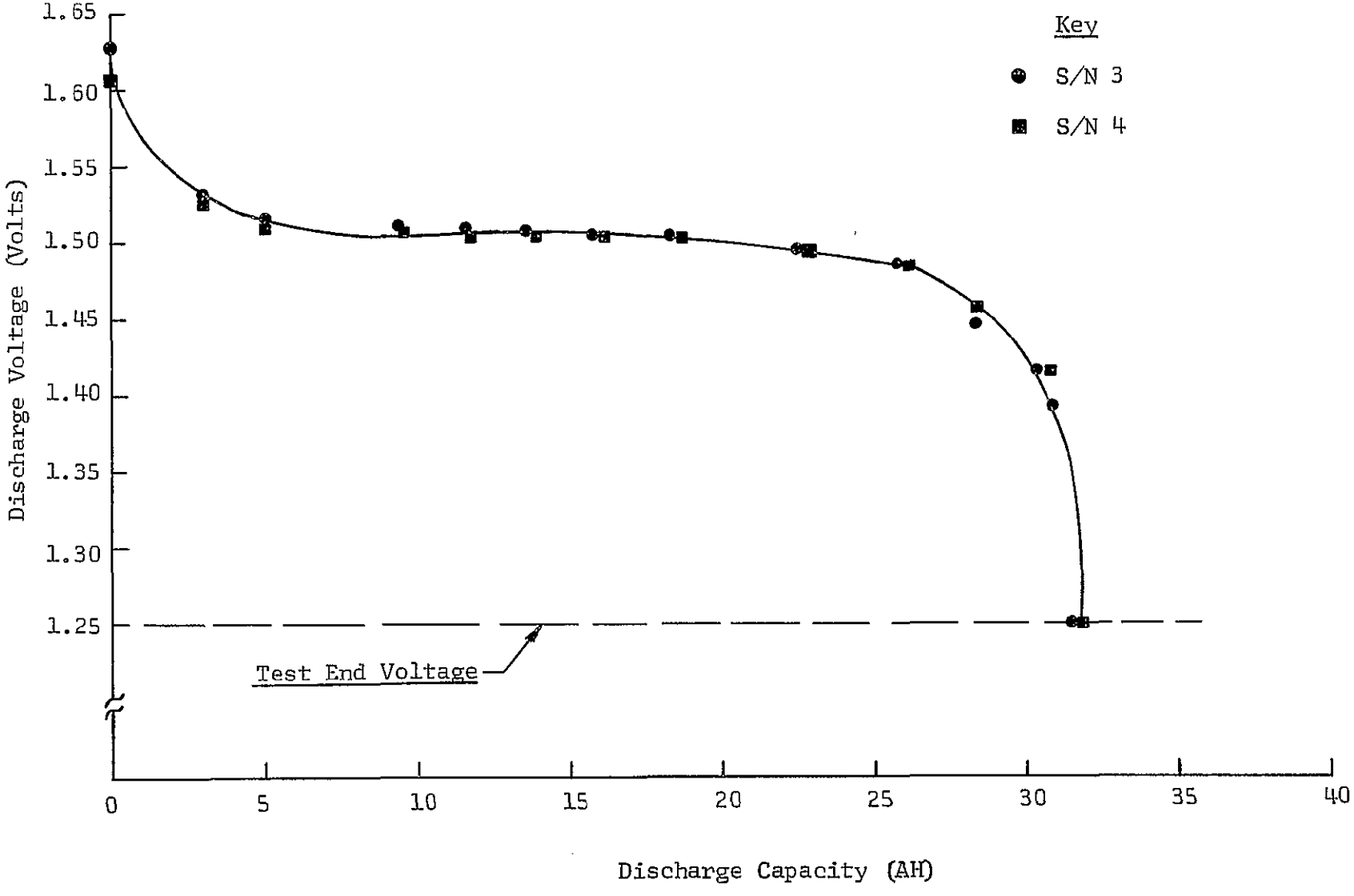


FIGURE 10

DISCHARGE VOLTAGE VS CAPACITY, MODEL 379
TYPE 2 HEAT STERILIZED CELLS
8 AMPS TO 1.25 V AT ROOM TEMPERATURE

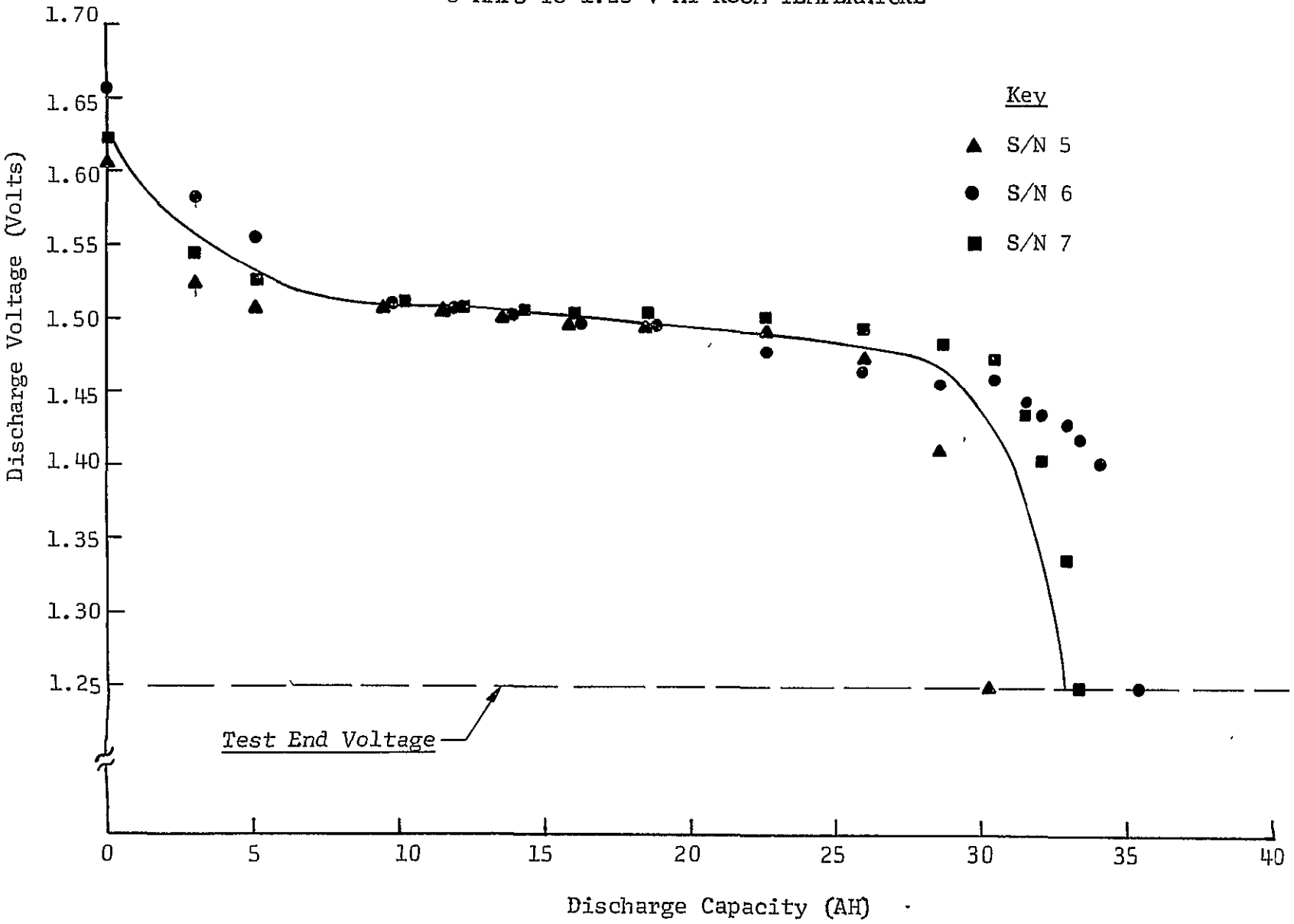


FIGURE 11

DISCHARGE VOLTAGE VS CAPACITY, MODEL 379
TYPE 3 HEAT STERILIZED CELLS
8 AMPS TO 1.25 V AT ROOM TEMPERATURE

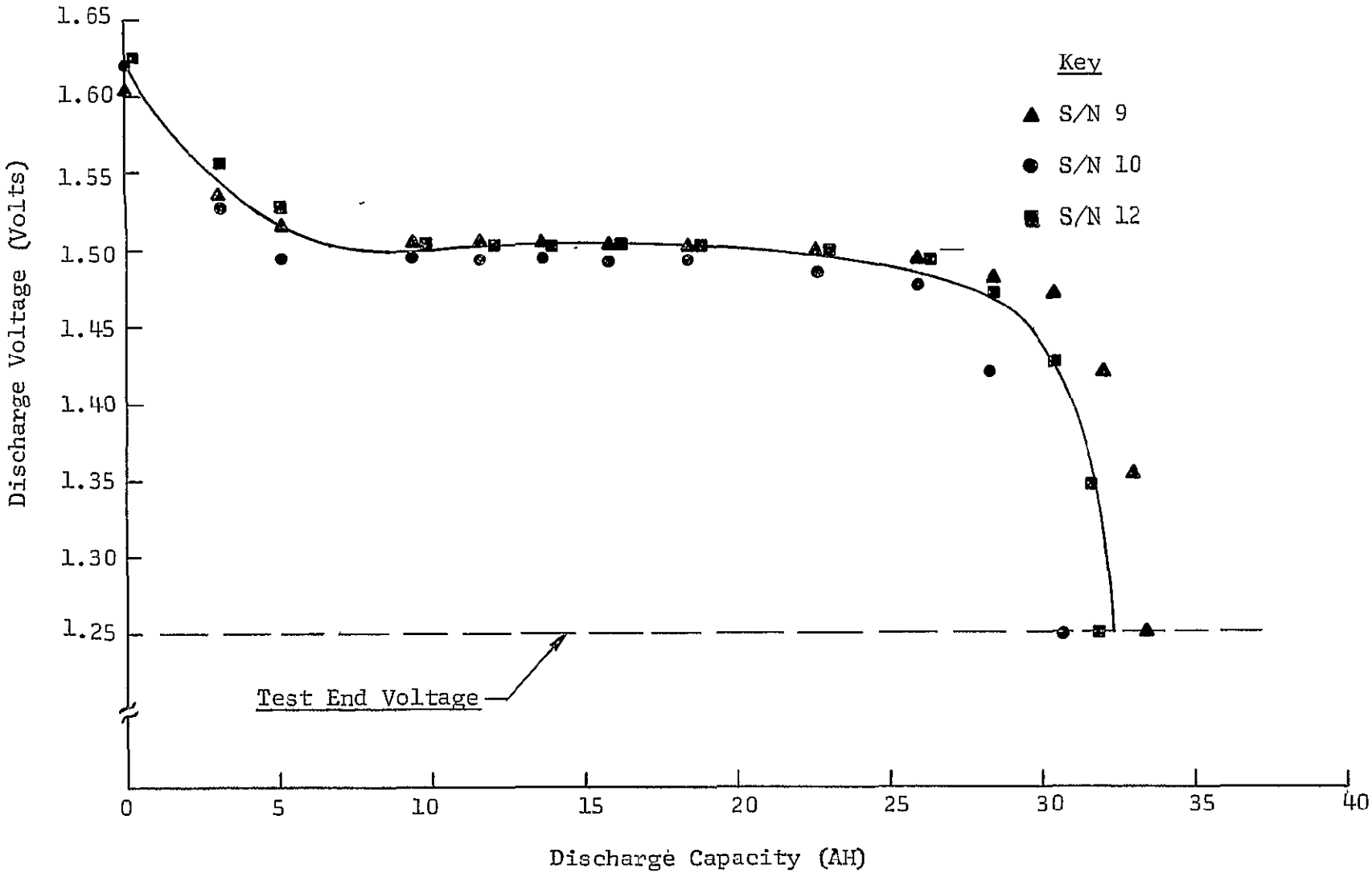


FIGURE 12

DISCHARGE VOLTAGE VS CAPACITY, MODEL 379
TYPE 4 HEAT STERILIZED CELLS
8 AMPS TO 1.25 V AT ROOM TEMPERATURE

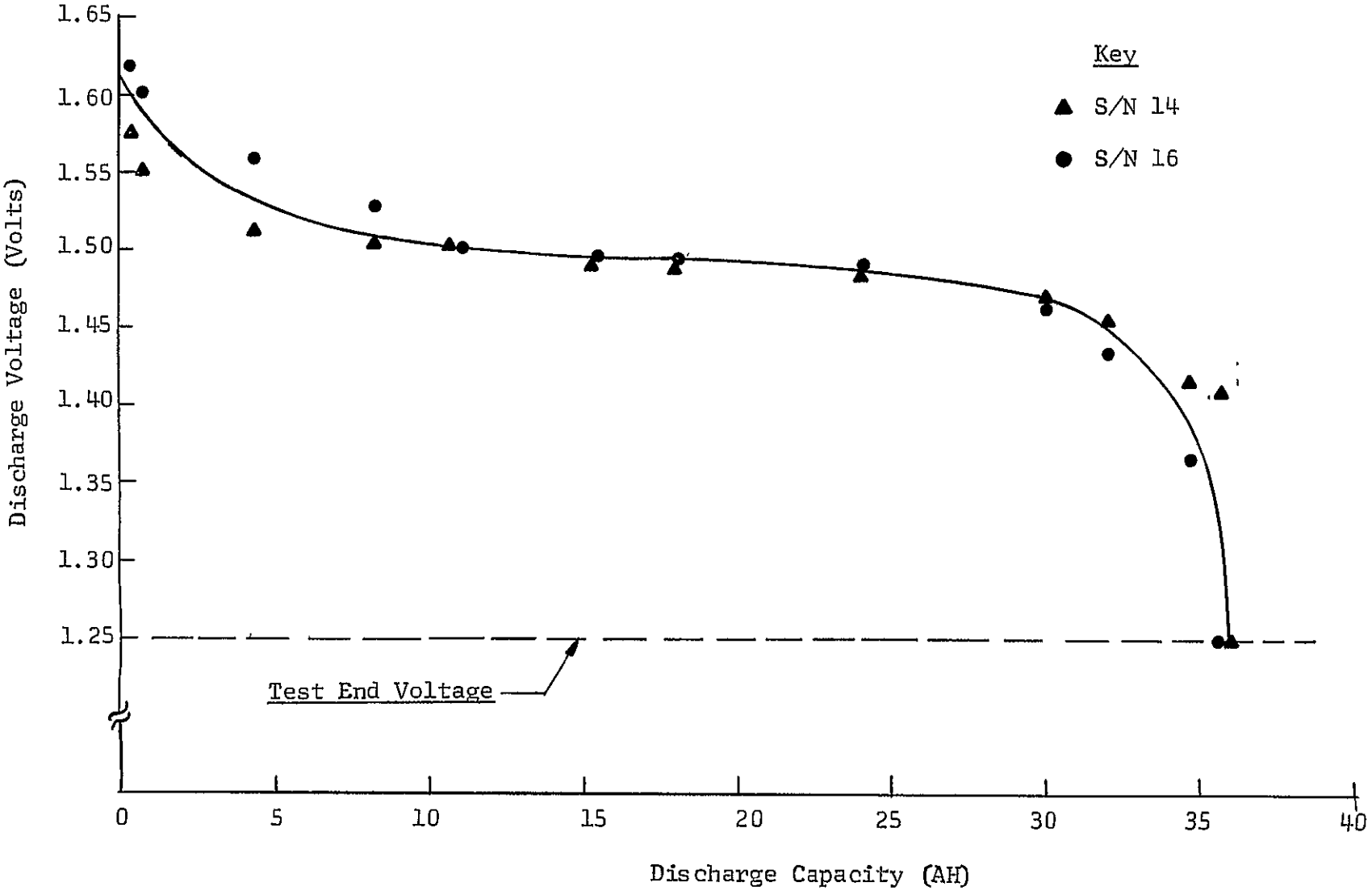
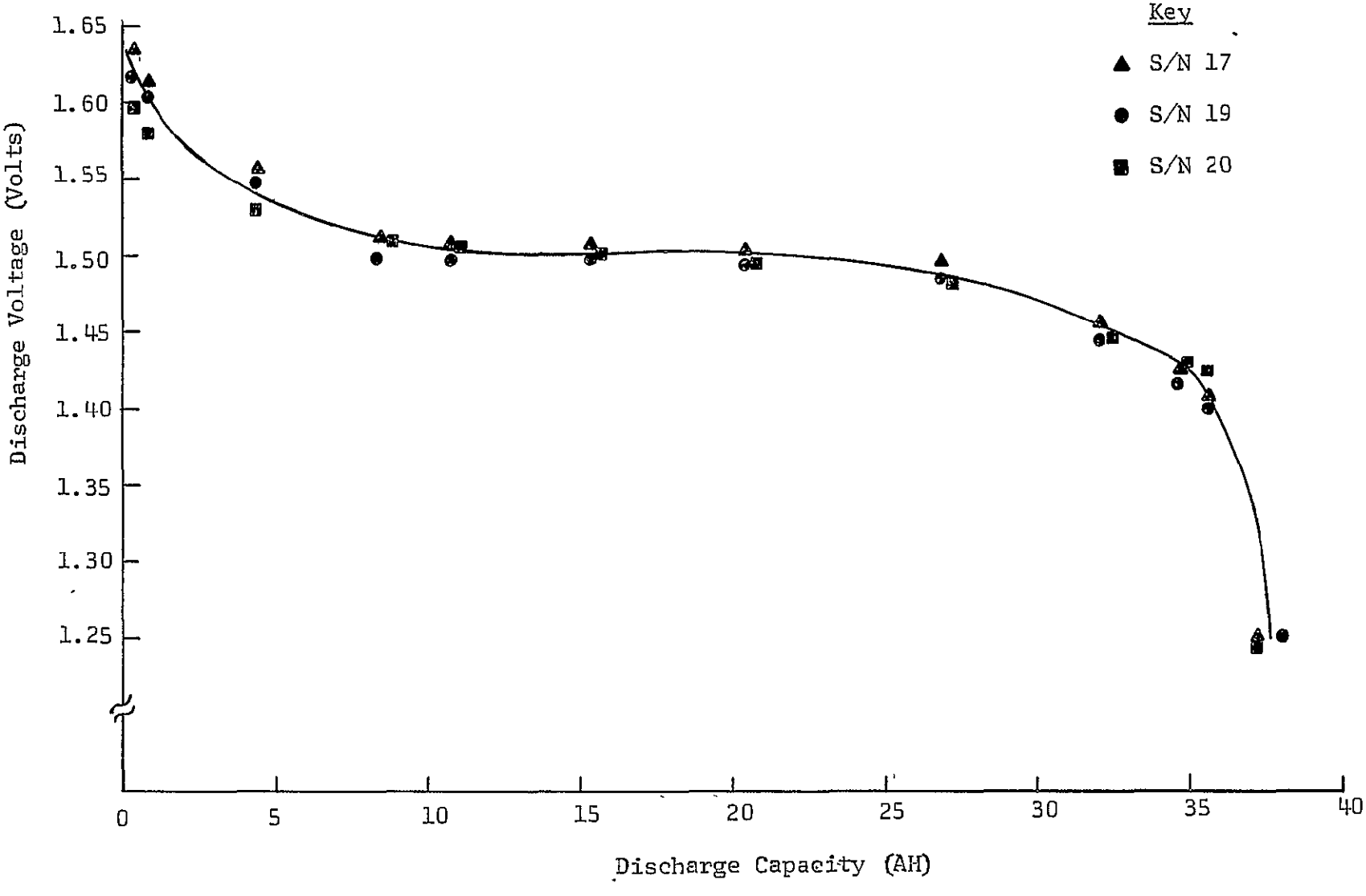


FIGURE 13

DISCHARGE VOLTAGE VS CAPACITY, MODEL 379
TYPE 5 HEAT STERILIZED CELLS
8 AMPS TO 1.25 V AT ROOM TEMPERATURE



five non-sterile cells representing five different designs. The above cells are now cycling at 50% depth on a 10 hour charge/2 hour discharge orbit at 25°C to reveal differences in capacity maintenance to 90 cycles.

C. Development Type "A" Cells. - One lot of 20 cells was released for production as development type "A" cells. Design type 379-3 was selected. Shipment was scheduled for April 14, 1969. The cover to jar epoxy seal formulation was changed from 100:5 to 100:3 parts (Dow DEN438EK85/DMP-30) to decrease seal crazing during heat sterilization on the basis of tests at EMED and JPL.

D. Development Type "B" Cells. - One lot of 20 cells design type 379-4 was released for production for delivery in early May 1969. These cells have been assembled through plate manufacturing stages.

E. Epoxy Seal Development. - While tensile strength tests have shown the epoxy system DEN438EK85/DMP30 (100:5) to be an excellent adhesive for PPO to PPO bonds, cell histories have shown this epoxy seal has a distinct tendency to craze during heat sterilization. For example, three of 15 Model 379 test cells leaked 3-6 gms water while the remaining 12 lost weight in the range of 1 to 2 gms. Cover to jar epoxy seal cracks were noted in all 15 cells. Evaluation of other epoxies has continued to eliminate this failure mode. Table XVII summarizes butt-joint strength tests on Epocast 31A (unfilled) and 31B (filled) epoxy, Furane Plastics, and a modification of the Dow DEN438EK85/DMP-30 system consisting of a reduction in catalyst DMP30 from 5 to 3 phr. Cure schedule was 24-36 hours at 25°C followed by 32 hours at 100°C with the samples immersed in

TABLE XVII

EPOXY BUTT JOINT STRENGTHS WITH PPO 534-801

Epoxy System (Mix Ratio)	No Sterilization				Sterilized 72 hrs. @ 135°C			
	Force to Break (lbs)	Area of Break (in^2)	Tensile Strength (psi)	Mean Strength (psi)	Force to Break (lbs)	Area to Break (in^2)	Tensile Strength (psi)	Mean Strength (psi)
31B/9216 (100:10)	150	.059	2530	3160	120	.058	2055	2250
	210	.059	3580		160	.057	2793	
	170	.058	2940		120	.057	2094	
	180	.057	3160		120	.058	2058	
	205	.057	3610					
31A/9216 (100:19)	80	.058	1370	1860	30	.059	510	970
	130	.058	2230		100	.057	1751	
	105	.059	1770		20	.059	341	
	130	.059	2210		40	.058	694	
	100	.059	1700		90	.059	1538	
DEN438EK85/ DMP30 (100:3)	110	.057	1920	3160	290	.058	5026	3260
	290	.058	4990		260	.058	4460	
	215	.057	3750		215	.057	3759	
	110	.058	1900		140	.058	2393	
	190	.058	3250		40	.059	678	
DEN438EK85/ DMP 30 (100:5)	240	.058	4130	2590	170	.058	2941	2770
	120	.058	2060		235	.059	4003	
	125	.058	2160		130	.058	2250	
	125	.057	2180		100	.058	1712	
	140	.057	2440		170	.058	2946	

NOTES:

- Cure - 24-36 hours @ room temperature + 32 hours @ 212°F in M40 KOH in a sealed bomb.

cell electrolyte in a sealed bomb. Under these conditions DEN 438EK85/DMP30 (100:3) yielded the highest tensile strength before and after heat sterilization 72 hours at 135°C and was selected as the sealant for the 20 type "A" cells.

F. Deflection of PPO 534-801 Jars Under Pressure. - Jars molded in PPO 534-801 and machined down to the height of the Model 379 cell case were sealed with the two test epoxies and then subjected without support to gradually increasing hydrostatic pressures. Figure 14 is a plot of the observed wall deflection to a pressure of 60 psig. These curves have been used to relate observed bulge to internal pressure of sealed cells without pressure gages. Pressures were increased to the burst point giving the following burst pressures before heat sterilization:

Epoxy Seal	Burst Pressure psig
Epocast 31B/9216 (100/10)	190, 175
Epocast 31A/9216 (100/19)	195, 180

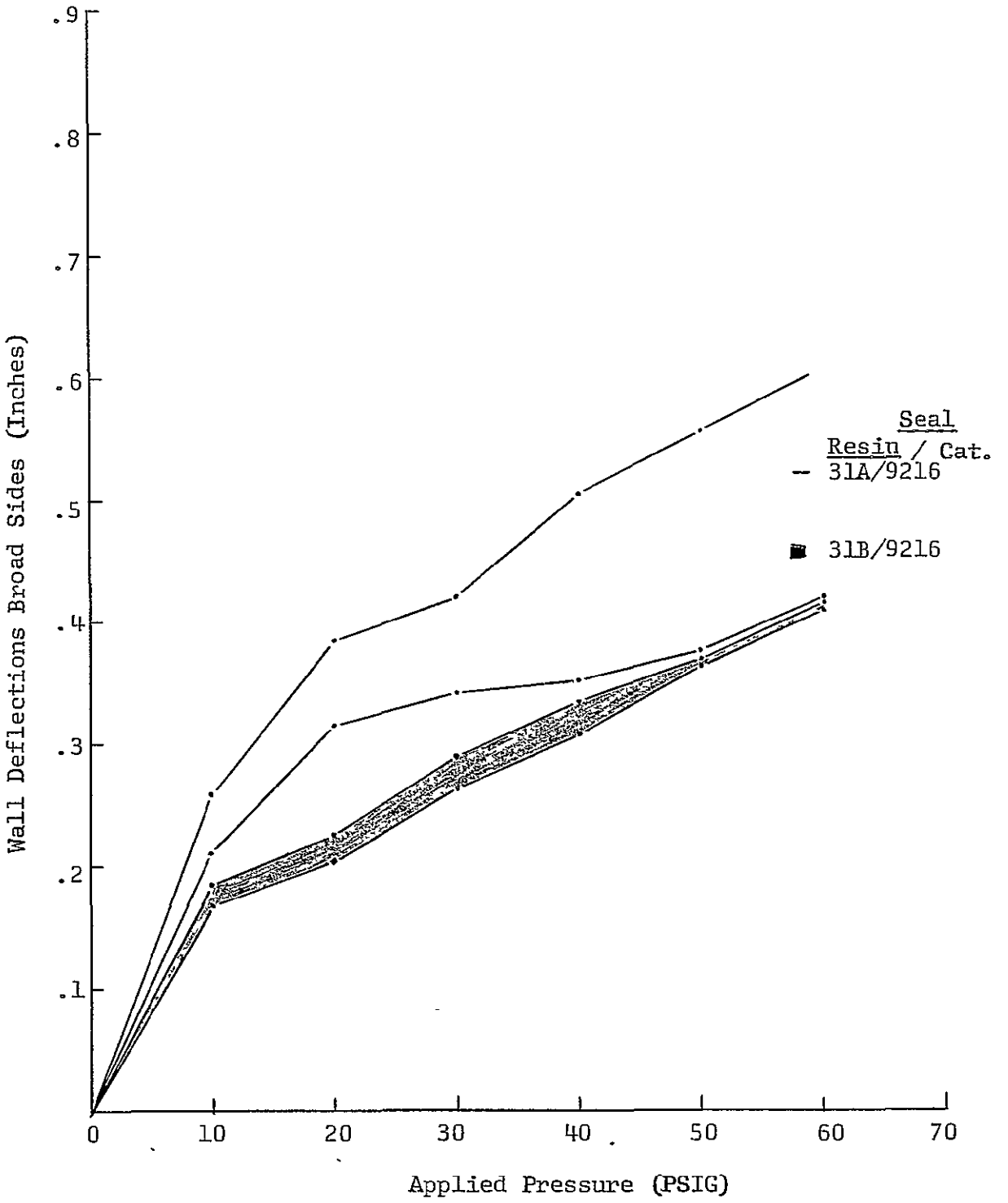
Failure mode was a combination of tensile shear of machined cover and epoxy peel; however, at 60 psig expected maximum pressures to safety factor is 3 for 1 for the epoxies above-before heat sterilization. Crazing of the DEN438EK85/DMP30 (100:5) seal during heat sterilization caused a pin hole leak at 10 psig and premature failure at 100 psig. Tests of the above seals after heat sterilization will be reported next quarter.

VI. CONCLUSIONS AND RECOMMENDATIONS

A. An epoxy platelock shows promise of improving vibration resistance and deceleration capability of wet heat sterilizable cells.

FIGURE 14

DEFLECTION OF MODEL 379 JAR WALLS
UNDER PRESSURE UNSUPPORTED



B. High impact-heat sterilizable positive plates reinforced with .010" zirconium sheet cores discharge at 17-21% lower efficiencies suggesting the need for greater than normal positive active material per rated ampere-hour in final cell designs No gas evolution occurs at the zirconium positives during wet heat sterilization but end of charge oxygen evolution occurs at 52% of theoretical capacity, considered premature at charge rates of 10 ma/in^2.

C. Heat shrinkable polyolefin tubing insulating plate leads has been shown to accelerate hydrogen evolution of zinc negative plates by a factor of 2-3 times at 140°F. Heat shrinkable teflon tubing is a satisfactory non-gassing replacement.

D. Design of 3,000 "g" 5 AH cell is complete. Non-sterile test cells are being assembled for impact tests from 2,000 to 4,000 "g" at JPL.

E. Twenty-seven cells, each of different design, have been assembled for a factorial experiment to determine effect on cycle life of electrolyte composition, wet thickness allowance for GX separator zinc oxide to silver active material ratio, and percent teflon in a dry sintered negative plate.

F. Silver analyses of GX separator at or near end of life in heat sterilized cells of two designs give values normalized by the factor milligrams of Ag per square inch of membrane (4th layer from positive) per ampere-hour total accumulative discharge capacity per month of wet life.

G. A plate fatigue tester has been designed, constructed, and tested to evaluate plate grid designs.

H. Cell design for 4,000 "g" high impact cell is complete. Positive and negative plate processes have been developed. Four nine plate cells are assembled for 2,000-4,000 "g" shock tests.

I. Twenty-five 25 AH cells, five each of five different designs, are on test to verify capability of 90 50% depth cycles after heat sterilization. Initial C/3 rate capacity measuring cycles show a loss of energy density of 6% due to 72 hours heat sterilization at 135°C at an output of 44 to 50 WH/lb of cell.

J. Two lots of 20 each 25 AH cells Model 379 type are in production for delivery to JPL in April and May 1969.

References: (1) E. K. Casani, Capsule System Advanced Development, CSAD Mission Model, 6 May 1968, JPL Report 760-20, p.9.

(2) Report for Fourth Quarter 1968 JPL Contract 951296, P.17, 18.

(3) IBID, p.25.

(4) IBID, p.30.

(5) IBID, p.32.